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Vol. 25 March 1, 1942 No. 5

SELENIUM RUBY GLASS*

By John D. Sullivan and Chester R. Austin

Abstract

This paper presents the results of an experimental study on the production of selenium ruby glass, particularly on the retention of the colorants during the melting and fining operations. Selenium, cadmium, and sulfur are necessary in the final glass to get a ruby color. Reducing conditions are conducive to the retention of a large percentage of the selenium in the glass, but when conditions are highly reducing, cadmium is eliminated almost completely. The writers believe that too little attention has been given in the past to the chemistry of the entire glass batch, and as a result operators and research workers have often failed to get a ruby glass not because the loss of selenium was too high but because they eliminated the cadmium by maintaining too strongly reducing conditions.

A balance must be maintained so that the batch and the atmosphere above it are sufficiently reducing to hold enough selenium in the melt but not so strongly reducing as to eliminate the cadmium. The use of silicon as a reducing agent is suggested.

Most of the work reported in this paper was done on soda-lime-silica glasses similar to those melted in continuous tanks for the production of machine-made ware. A glass of this composition, containing 0.03% of selenium, 0.06% of cadmium, and 0.03% of sulfur, will produce a ruby color. The writers have found that if the cadmium is added as cadmium sulfide the final glass will have sufficient sulfur for color development.

An arrested-cooling procedure for developing color is described briefly.

I. Introduction

This paper reports the results of experimental work on factors involved in the production of selenium ruby glass. Consideration is given more specifically to the retention of the ruby-developing colorants in the glass during the melting and fining processes. There is fairly general agreement among operators and research workers that the selenium ruby color is due to a precipitated compound comprising selenium, cadmium, and sulfur. Some evidence also substantiates the belief that the color is due to cadmium sulfoselenide. The theory of ruby-color formation is not particularly stressed in this paper, but the authors hope at a later date to report on a research program, already substantially completed, on this subject.

Most commercial selenium ruby glass, such as that used in signal glass, has been made with a base glass which contains appreciable amounts of zinc oxide. A typical composition is as follows:

Ingredient	(%)	Ingredient	(%)
SiO_2	69.2	ZnO	9.4
Na_2O	16.0	B_2O_3	0.5
K_2O	3.1	$Ca_3(PO_4)_2$	1.4
Al_2O_3	0.4	Total	100.0

Virtually all commercial selenium ruby glasses are made in pots or in small day tanks. It is rather well established that selenium losses are less in crucible or pot melting than in the open-flame type of furnace.

Although most of the experiments reported here were conducted on soda-lime-silica glasses, enough work was done with zinc oxide-containing glasses to show that the same general factors which involve the retention of colorants in manufacture hold.

In the customary commercial methods of making selenium ruby glasses, 1% or more, by weight, of selenium, usually in metallic form, is added to the batch. Cadmium and sulfur are usually added as cadmium sulfide, frequently in amounts of 0.8% or more, by weight, of the raw batch. The analyses of commercial selenium ruby glasses showed, however, that the selenium con-

* Presented at the Glass Division Autumn Meeting, Conneaut Lake, Pa., September 13, 1941. Received July 10, 1941.

tent seldom exceeded 0.20% and was frequently much lower. The work reported in this paper, moreover, showed that satisfactory ruby glasses can be produced with the selenium content as low as 0.03 or 0.04%. A large percentage of the selenium is lost in the melting and fining operations; an appreciable excess, therefore, must be present in the batch in order to have enough in the final glass. This statement is true regardless of the form in which the selenium is added. It was added in various forms in this investigation, but the retention usually was substantially as good, and frequently better, when metallic selenium was employed than when other forms were used. For this reason, unless stated to the contrary, the metallic form was used in the work here reported. In small-scale tests, however, P_2Se_5 and NaCNSe effected better retention than the metallic form.

As will be shown in section II (7A), selenium, cadmium, and sulfur must be present in the final glass to get a ruby color. Conditions which are conducive to retaining selenium in the melt may effect a substantially complete loss of the cadmium, and glasses containing even more than 0.5% of selenium will not produce a ruby color if cadmium is absent or too low. Past experimenters and operators appear to have frequently neglected the consideration of retaining cadmium in the melt, and they have also incorrectly placed the blame for failure to develop color on the loss of selenium rather than on the loss of cadmium. The ruby glasses of the selenium-cadmium-sulfur type are referred to here as "selenium ruby" to conform to the usual trade practice.

II. Experimental Work

The following information is given to clarify and to simplify the presentation of the experimental work, the data, and the discussion. It will hold in all cases unless specifically stated to the contrary.

(1) Base Glass

The base glass fell in the following composition ranges:

Ingredient	Weight (%)
SiO_2	69–74
Al_2O_3	1– 4
$Na_2O + K_2O$	13–16
$CaO + MgO$	7–13

(2) Fuel and Gaseous Atmosphere

Natural gas was used both in the crucible and in the day-tank melts. A reducing atmosphere above the melt, as used in this paper, indicates one which contains no free oxygen and from 1 to 4% of free carbon monoxide, CO.

(3) Crucibles and Day Tank

Crucible melts were made in gas-fired pot-type furnaces. The crucibles had a batch capacity of 500 or 3000 gm. The 500-gm. batch crucibles were the fireclay type, and the latter were the mullite type.

The day tank most widely used (Fig. 1) was lined with mullite-type flux blocks. The capacity was about 500 lb. of melt. A few tests, however, were made in a 600-lb. capacity tank.

(4) Development of Color

The color was developed by "fire striking" in some instances and in others by an arrested-cooling procedure. As this discussion is devoted primarily to the chemistry of retention of the color ingredients, the particular method in which the color was developed is of secondary importance. In section II (9), an arrested-cooling procedure, developed in this study, is described briefly.

FIG. 1.—Day tank.

(5) Oxidizing vs. Reducing Conditions

Even in crucible melts where conditions are most favorable, with 1.0% of selenium in the batch, the retention was practically zero unless reducing conditions were maintained. If no reducing agent was added to the melt and if the gaseous atmosphere was oxidizing, that is, if it contained no free carbon monoxide, virtually all of the selenium was lost. It may be possible to retain sufficient selenium in the crucible melts by maintaining a reducing gaseous atmosphere; in day-tank melts, however, it is necessary to have a reducing agent in the melt and to maintain a reducing atmosphere or one only slightly on the oxidizing side.

The following crucible tests show the effect of the addition of carbon (lampblack) on the retention of selenium in the melt. In each test, the batches were charged at 2700°F., held in the furnace for 3 hours, and then removed from the furnace. The atmosphere was reducing.

Melt	Se in final glass (%)
Base batch + 1% Se + 0.6% CdS	0.013
Base batch + 1% Se + 0.6% CdS + 1.0% C	0.69

(*A*) *Day-Tank Tests:* A description of two tests is given to show how rapidly selenium was lost in a day tank even though the atmosphere above the melt was reducing.

Test A: The ingredients, which were melted in a day tank on a schedule given in Fig. 2, and the calculated

oxide composition of the base glass, excluding colorants, are as follows:

Ingredient	(lb.)	Ingredient	(%)
Sand	355	SiO_2	72.5
Soda ash	114	Na_2O	12.5
Burned dolomitic lime	62	K_2O	1.5
Feldspar	68	CaO	6.6
Selenium	6	MgO	4.4
Cadmium sulfide	3.6	Al_2O_3	2.5
			100.0

Additions of 1.0% of selenium and 0.6% of cadmium sulfide were made to the raw batch, and the tank temperature was about 1850°F. at the start of charging. During the first 8 hours of the melt, the furnace atmosphere was maintained at about 3 to 4% of carbon monoxide and no free oxygen. During the last period, the atmosphere contained 1 to 2% of carbon monoxide.

The results of the chemical analyses for selenium, which were made on trials drawn during the melt, are also plotted in Fig. 2. The final selenium content, however, was too low to yield a ruby color.

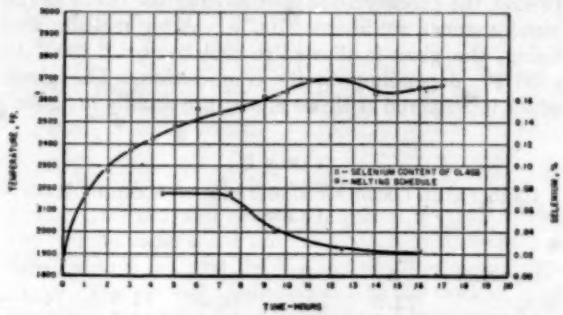

FIG. 2.—Melting schedule and corresponding selenium content of melt.

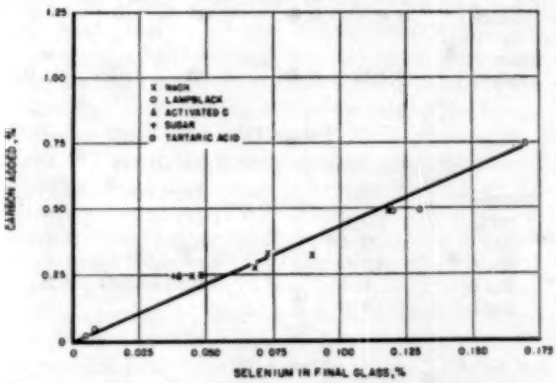

FIG. 3.—Effect of amount and form of carbon on selenium retention; 0.17% of Se in batch.

Test B: This test was run under substantially identical conditions of test A, but sodium silicate containing 22.9% of Na_2O and 74.4% of SiO_2 replaced all of the soda ash, and the remainder of the batch was adjusted to give the same oxide composition as in test A. The final selenium content was 0.04%, which was about twice that in the previous test, sufficient to develop a ruby color.

In the tests A and B, no reducing agents were added to the melt. Although crucible or pot melting might be carried out successfully without the use of reducing agents in the melt, it appears that some reducing agent sholud be present in day tanks or in full-size continuous melting furnaces.

(6) Effect of Various Reducing Agents on Selenium Retention

Data are given in Fig. 3 on the effect of various reducing agents on the retention of selenium in crucible melts. When strong reducing agents were present, even in moderate amounts, a substantial percentage of the selenium remained.

(7) Effect of Reducing Agents on Retention of Cadmium

A 400-lb. melt in a day tank employed an addition of 1.0% of selenium, 0.6% of cadmium sulfide, and 1.0% of carbon. The final glass did not produce a ruby color by any of the ordinary heat-treatment methods, but it contained 0.59% of selenium. The use of carbon effected the retention of selenium, but it caused the loss of some ingredient essential to the formation of the ruby color. Chemical analysis proved that the glass had 0.13% of total sulfur in addition to 0.59% of selenium but 0.00% of cadmium. The use of carbon, therefore, while effective in retaining selenium in the melt, caused the loss of all of the cadmium, and the resultant glass was not a ruby. A large amount of selenium in the final glass has a tendency to give a brown cast to the glass, and carbon has the same tendency.

Data are given in Table I on four tests, in three of which a reducing agent was added. Test No. 1 is that just described. Tests Nos. 1, 2, and 3 did not produce a ruby color, whereas No. 4, which was melted without a reducing agent, resulted in ruby color. It was also the only one to contain cadmium. Many other tests could be reported, but these given are typical and show why, in the past, many operators and research workers have erroneously believed that because selenium was lost they were not getting a ruby glass. The chemistry of the entire system involved must be considered.

TABLE I
CHEMICAL ANALYSES OF GLASSES

Glass No.	Added ingredients (%)			Per cent in final glass		
	Selenium	CdS	Carbon	Selenium	Total sulfur	Cadmium
1	1.00	0.6	1.0	0.59	0.13	Nil
2	0.12	0.6	0.25	0.06	0.13	"
3	0.34	0.6	0.17*	0.04	0.05	"
4	0.60	0.6		0.03	0.03	0.06

* Added as NaCN.

Experiments on the addition of cadmium in various forms indicate that cadmium sulfide is as effective as any form in respect to cadmium retention.

(A) Other Reducing Agents: The study of the formation of selenium ruby glass therefore resolved itself to determining the conditions which are sufficiently reducing to effect the retention of enough selenium but

not so harsh as to cause the loss of too much cadmium. The minimum amount of cadmium required in the glass to get a ruby cannot be stated definitely, but it is approximately 0.06%. The chemical analyses indicated that the total sulfur was present in adequate amounts in all cases, but the methods of analysis unfortunately do not differentiate the various forms in which the sulfur may be present.

Carbon, for example, in the form of lampblack has the disadvantage that it tends to discolor the glass. A study was made in 3000-gm. crucibles of the following general classes of "mild" reducing agents: (1) sulfur and sulfur-containing compounds, (2) silicon and silicon-containing compounds, and (3) lignin products. In all instances, 1.0% of selenium and 0.6% of cadmium sulfide were employed, and the batches were added to crucibles at 2700°F., melted, and fined for 4 hours at that temperature. A reducing atmosphere was maintained.

The results with sulfur were not particularly encouraging, and 4% was necessary to retain 0.04% of selenium in the final glass.

The use of silicon, 60-mesh in size and of metallurgical grade, gave encouraging results, and as little as 0.05% of silicon in a batch containing 1.0% of selenium and 0.6% of cadmium sulfide resulted in a sufficient quantity of selenium, cadmium, and sulfur in the final melt. An addition of 1.0% of silicon yielded a glass containing 0.77% of selenium. Silicon carbide and calcium silicide also resulted in a high-selenium retention, but no recommendation can be made as to their use because the purpose of the tests in which they were used was solely to study retention.

Lignin products, like carbon, had the disadvantage of imparting a brown color to the glass.

(8) Other Factors on Selenium Retention

(*A*) *Variations in Composition:* Five glasses of varying composition but of the soda-lime-silica type were melted in crucibles without the addition of reducing agents but with a reducing atmosphere. The results given in Table II show that the selenium retention was practically the same in all cases.

Zinc Oxide: One melt containing an appreciable amount of zinc oxide showed that, although the final glass of the composition given in Table III contained 0.32% of selenium, it did not develop a ruby color except by extremely slow cooling in the crucible.

High Potash: Several melts were made to determine the effect of replacing Na_2O by K_2O. Frequent references in the literature advocate the use of K_2O in selenium-ruby glasses. The calculated oxide compositions of the glasses studied and of the soda-lime-silica base glass are given in Table IV. Apparently no benefit was derived from the substitution of K_2O for Na_2O in a base glass of the composition used in this work. The potash glasses had an extremely short working range and were difficult to "gather" by the usual method.

(*B*) *Effect of Additions of Iron, Cobalt, and Nickel Compounds:* The additions of small amounts of various compounds of iron, cobalt, and nickel to a selenium-containing batch considerably increased the retention of selenium. As shown in Table V, from four to six times as much selenium was retained when iron, cobalt, or nickel were added. Compound formation may account for this increased retention of selenium. These observed facts are of fundamental interest and may be particularly significant in decolorizing glass by selenium.

The additions given in Table V are not recommended for producing a ruby glass, and the only purpose of including these data is to show the effect of the additions on selenium retention.

(9) Color Development by Arrested Cooling

Commercial selenium-containing ruby glasses as formed ordinarily are not ruby colored, and the color is developed by a reheating operation after the ware is formed or is in the mold. The present research showed that the ruby color can be developed in soda-lime-silica glasses by a simple arrested-cooling operation, such as that commonly employed in the metallurgical field in the heat-treatment of metals and alloys. Briefly, the process consists of melting the batch in the usual manner at about 2700°F. After melting and fining, the glass is allowed to cool to about 2200° to 2300°F., depending on its composition. The glass, which is delivered at this temperature usually in "gobs"

TABLE II

CALCULATED OXIDE COMPOSITIONS OF VARIOUS BASE GLASSES

Ingredient	Per cent in glass				
	A	D	F	G	H
SiO_2	72.5	72.5	72.5	72.5	74.4
Na_2O	12.5	15.0	13.5	14.0	12.8
K_2O	1.5	1.5	1.5	1.5	1.5
CaO + MgO	11.0	8.5	10.0	9.5	8.7
Al_2O_3	2.5	2.5	2.5	2.5	2.6
Se (added)	0.6	0.6	0.6	0.6	0.6
Se in final glass (by analysis)	0.026	0.03	0.020	0.022	0.025

TABLE III

COMPOSITION OF ZINC-BASE GLASS

Ingredient	(%)	Ingredient	(%)
SiO_2	69.9	CaO	3.2
Al_2O_3	2.4	MgO	2.1
Na_2O	12.0	Se (added)	0.6
K_2O	1.4	CdS (added)	0.6
ZnO	9.0		

TABLE IV

COMPOSITION OF HIGH-POTASH GLASSES

Ingredient	Per cent in glass			
	KB	KC	KD	A*
SiO_2	68.1	74.2	66.6	72.5
Na_2O	0.4	0.4	0.4	12.5
K_2O	18.7	11.4	20.4	1.5
Al_2O_3	2.3	2.5	2.3	2.5
MgO + CaO	10.5	11.5	10.3	11.0
Se (added)	0.8	0.8	0.8	0.8
CdS (added)	0.6	0.6	0.6	0.6

* Soda-lime-silica base glass used previously.

of sufficient size to form the desired article, is then subjected to an arrested cooling in the temperature range of 1400° to 2000°F. The length of time the glass is held in this range to develop the color depends on the temperature. At 1400°F., only 5 to 10 seconds is necessary, whereas at 2100°F. the time is about 1 minute. After the arrested cooling procedure, during which the ruby color develops, the molten mass of glass is formed into the desired shape by some standard method. This process leads to a more uniform color throughout the piece than that obtained by fire striking.

TABLE V

EFFECT OF ADDITIONS OF IRON, COBALT, AND NICKEL COMPOUNDS ON SELENIUM RETENTION

Batch	Compound added	(%)	Selenium retained in final melt (%)†
A*	$CoCO_3$	0.10	0.25
A	Fe	.10	.22
A	Fe	.50	.20
A	Fe_2O_3	.13	.23
A	$FeCO_3$	.10	.24
A	$FeC_2O_4.2H_2O$‡	.13+	.30
A	NiO	.10	.22
A	None		.05

* See Table II for composition.
† 0.6% of selenium added to batch throughout.
‡ 0.2% of C also added.

III. Discussion

The chief purpose of this discussion is to show that the entire chemistry of the glass batch must be kept in mind in any work on the production of selenium ruby glass. The present studies as well as the work of other experimenters have demonstrated that oxidizing conditions are conducive to high selenium losses. In manufacturing signal glass, for example, 1 to 1.5% of selenium is usually added to the pots in order to have enough in the final glass to get the desired color. Selenium is expensive, and the production of cheap glass requires that its loss be not too great. This problem is aggravated further in making a selenium ruby glass in a tank because the losses are higher than when pots are used. Even if the gases over the melt are reducing, the selenium losses are great because of the high vapor pressure of selenium or of one of its compounds, possibly the oxide. The rapid sweeping of combustion gases over the melt intensifies the removal of selenium.

It is necessary, therefore, to add something to the batch to hold the selenium in the melt. Oxidizing agents are fatal because selenium dioxide, SeO_2, has a high vapor pressure and sublimes at as low as 317°C. (603°F.). As shown in this work, reducing agents are effective in holding large percentages of the selenium in the melt, and as much as 78% has been thus retained under certain conditions. Strong reducing agents, however, such as carbon, unfortunately eliminate cadmium, and as a result a ruby color is not obtained. The retort process for obtaining cadmium and zinc, which is familiar to metallurgists, explains why cadmium would be lost when strong reducing agents are present in the melt. If carbon-containing reducing agents are present in appreciable amounts, they discolor the glass, usually giving a brownish cast.

It is clear, therefore, that controlled reducing conditions within the melt are necessary to retain both selenium and cadmium in the melt in requisite amounts. Definite recommendations cannot be made at this time, but the use of metallic silicon, which forms silicon dioxide on oxidation, seems to be a promising lead. Other experimenters may find something better, and it is hoped that others will conduct fundamental studies on the retention of the three necessary ingredients selenium, cadmium, and sulfur. With a knowledge of what must be accomplished, the goal should be reached.

It has been demonstrated that a ruby glass can be made in day tanks with soda-lime-silica base glasses, similar to those used in machine-made ware. It also appears possible to make such selenium ruby glasses in continuous tanks and to form the ware by machine methods.

IV. Analytical Methods

The selenium was determined by the distillation method of Pavlish and Silverthorn.[1]

The cadmium was determined by dissolving the sample in a mixture of hydrochloric, nitric, sulfuric, and hydrofluoric acids and fuming, diluting, and precipitating the cadmium as CdS with hydrogen sulfide. The precipitate was contaminated with selenium; so it was dissolved in nitric and sulfuric acids, and the selenium was precipitated with sulfur dioxide. After filtration, the cadmium was again precipitated as sulfide and subsequently converted to cadmium sulfate, $CdSO_4$, and weighed.

The sulfur was determined by fusing the sample with a mixture of sodium carbonate and potassium nitrate, leaching, acidifying with hydrochloric acid, and boiling to reduce selenates, and precipitating with barium chloride as $BaSO_4$. The precipitate was then treated with hydrofluoric and sulfuric acids to remove silica, ignited, and weighed.

Acknowledgment

The permission of the Selenium Development Committee to publish the results of this investigation and the assistance of S. J. Brooks in carrying out the laboratory work are gratefully acknowledged.

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[1] A. E. Pavlish and R. W. Silverthorn, "Analysis of Selenium in Glass," *Jour. Amer. Ceram. Soc.*, **23** [4] 116–18 (1940).

SELENIUM BLACK GLASS*

By Chester R. Austin and John D. Sullivan

Abstract

A black glass was made by melting a soda-lime-silica glass under a reducing atmosphere with the addition of 0.6% of selenium and 0.1% of cobalt carbonate, $CoCO_3$. The light absorption of a specimen about 0.01 inch thick in the range of 400 to 750 mμ was superior to that of commercial black glass. The maximum transmission was 27% at 750 mμ. The use of nickel and iron oxides is less effective than cobalt oxide for obtaining selenium black glass.

I. Introduction

In a general investigation on the role and uses of selenium in glass, the production of various colored products was studied. One of the most interesting was black glass of the soda-lime-silica type with only relatively small additions of colorants. The present method of manufacturing black glasses usually involves the addition of substantial amounts of colorants, such as oxides or sulfide of iron or oxides of manganese and iron and oxides of nickel and cobalt. Inasmuch as these additions often amount to 10 to 15% of the entire batch, the working properties of the glass are often affected adversely. It would appear, therefore, that a batch composition and a process to obtain a black glass containing only minor amounts of color-producing ingredients would be useful.

II. Experimental Work

The authors have discussed the experimental procedure and results in producing a selenium ruby glass,[1] and as the general operations used were the same, they are not repeated here. All of the results given here are on base glasses in the following composition ranges:

Ingredient	Weight (%)
SiO_2	69–74
Al_2O_3	1– 4
$Na_2O + K_2O$	13–16
$CaO + MgO$	7–13

Most of the work was done with a base glass of the following composition:

Ingredient	(%)	Ingredient	(%)
SiO_2	72.5	CaO	6.6
Na_2O	12.5	MgO	4.4
K_2O	1.5	Al_2O_3	2.5

All of the experiments were made in crucibles up to 3000-gm. batch capacity with natural gas as the fuel. The term "reducing atmosphere" above the melt as used in this paper indicates one which contains no free oxygen and from 1 to 4% of free carbon monoxide, CO. The importance of reducing conditions in the retention of selenium has been pointed out in the previous paper.[1] In the present work on black glass, experiments were made with oxidizing and reducing atmospheres. Reducing agents were not added to the batch in most cases, but they are even more effective in retaining selenium than reducing atmospheres.[1]

(1) Effective Additants

The addition of oxides or oxide-forming compounds of cobalt, nickel, or iron with selenium was effective in producing a black glass. Cobalt was the most effective, and the best black was obtained from a batch containing 0.6% of selenium and 0.1% of cobalt carbonate. Larger amounts of selenium were not necessary. The best procedure in making the black glass was to melt at about 2700°F., cool, and form.

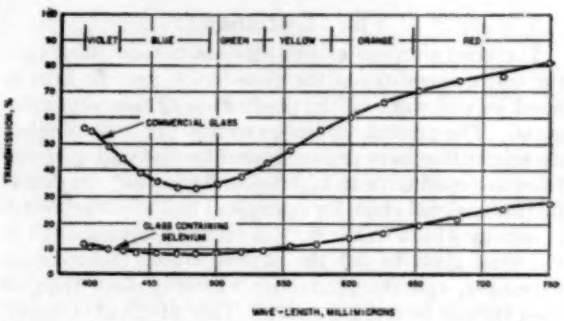

Fig. 1.—Transmission curves; specimen 0.01 in. thick.

Figure 1 gives transmission curves of black glass, which was made from a batch containing 0.6% of selenium and 0.1% of cobalt carbonate, and of a commercial black glass in the wave-length range of about 400 to 750 mμ. The specimens were approximately 0.01 in. thick. The experimental glass contained 0.18% of Se. The selenium-cobalt glass was superior in absorption in the "black range." Its maximum transmission up to 750 mμ was 27%, whereas the minimum transmission for the commercial glass was 34% at 475 mμ, and at 750 mμ, the transmission was 82%.

(2) Effect of Amount of Selenium Added

In the crucible melts with reducing atmospheres of the type used, it appeared necessary to use about 0.6% of selenium in the batch. Additions of 0.4% gave a black color but one not so opaque as with the larger addition. In this connection, however, the retention of selenium in the final melt rather than the amount

* Received October 20, 1941.

[1] J. D. Sullivan and C. R. Austin, "Selenium Ruby Glass," this issue, pp. 123–27.

added in the raw batch is the governing factor. On the basis of the work done, it would appear to be necessary to have at least 0.15 to 0.20% of selenium in the final glass. This retention might be effected by the addition of a small amount of a reducing agent to the batch even if less than 0.6% of selenium is used initially. If the atmosphere is oxidizing enough, selenium is not retained even when 0.6% is added. In a test maintaining 3% of free oxygen in the atmosphere above the melt and with no reducing agent in the batch, the final glass had 0.02% of selenium. This glass was not black, but due to the cobalt, it had a blue color.

The work also indicated that 0.05% of $CoCO_3$ was not enough to produce a black color when 0.4% of selenium was used in the batch.

(3) Iron and Nickel Oxides

Oxides of iron and nickel were not so effective as cobalt. Nickel oxide yielded an opaque black glass, but the color had a brownish-black cast. It also appeared necessary to add more Ni_2O_3 than CoO. The color from iron additions likewise was on the brownish-black side, and more Fe_2O_3 than CoO was necessary.

III. Discussion

The results indicate that it is possible to get an excellent black glass with only minor additions of colorants. Less than 0.2% of selenium and less than 0.1% of cobalt (0.1% of $CoCO_3$ ≏ 0.05% of Co in batch) would seem to be necessary in the final melt to give a black color. The batch must be melted under reducing conditions or with a reducing agent in the batch. The small amount of colorant does not affect the working properties of the glass. There should be commercial applications both in pressed ware and in structural type of glass.

Acknowledgment

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ZIRCONIUM OXIDE AND THORIUM OXIDE IN CERAMICS*

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Abstract

The properties of the most important zirconium compounds are correlated to the main ceramic applications of these compounds, namely, refractories, ceramic bodies, binders, cements, glazes, glasses, and enamels. A brief survey of the information available on the systems $MgO–ZrO_2$, $TiO_2–ZrO_2$, $SiO_2–Na_2O–ZrO_2$, and several other systems is included. The more specific ceramic properties are summarized in short descriptions of the applications to each single ceramic field. The ceramic uses of thorium oxide are listed.

I. Introduction

Engineering is predominantly responsible for the progress of the ceramic industries. Energy-saving mass production has not only increased the importance of these industries but has also financed ceramic research and has stimulated new pioneer branches of ceramics. This present development might be called the third period of ceramic knowledge. The first is the hit-and-miss period of valuable traditions which covers several thousand years, and, in many branches of ceramics, it reaches well into the present century. The second period is closely related to the chemical achievements of the 19th century. Chemistry and physics first analyzed the traditional experience of the preceding period, and then, rapidly and systematically, introduced elements, compounds, processes, and research methods to achieve an admirable expansion. The development of optical glasses and of ceramic colors is an example of this period.

In the present period, chemical and physical research is continuing as the faithful servant of ceramic engineering. Apart from the perpetual value of continued fundamental research, another lasting task of chemical and physical investigations of immediate service to these industries is the utilization of raw materials of local importance or of low cost and the development of rarer materials.

Are all rare materials definitely applicable to the ceramic industry, or must they be excluded from it? Can it be that rarity, an unchangeable attribute of certain forms of matter, is sometimes confused with the fluctuating attributes of availability and cost? A chart of the occurrence of some elements in the earth's crust (Fig. 1) proves that there is a tendency to consider some elements as being rare which do not belong in this classification. The height of the twelve steps on the chart symbolizes the occurrence of a number of elements on a logarithmic scale. The lowest step represents an occurrence of 10^{-10}%, the highest an occurrence of 10%.

Zirconium, thorium, cerium, and the rare earths are so-called rarer materials. These elements, which can be useful for ceramic ware, are the subject of this discussion. None will be found on any of the lower steps; rather, all of them occur in an abundance of $^1/_{1000}$ to $^1/_{100}$%. Beryllium precedes such familiar ele-

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ments as arsenic, tin, and antimony; thorium also precedes these, as well as lead; and cerium and zirconium, which are five times more frequent than zinc and of the order of 100 times more abundant than tin.

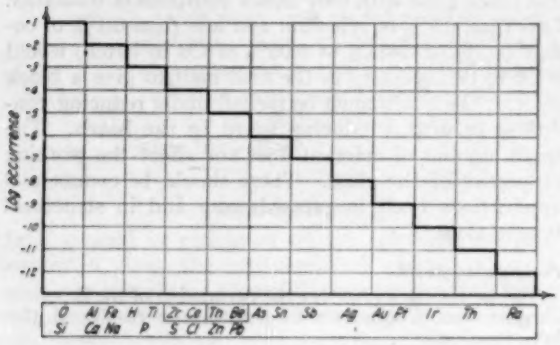

FIG. 1.—Occurrence of some elements in the earth's crust (according to Clark and Washington).

Some of the so-called rarer materials, therefore, are not rare; they are (or were) not so easily available or so cheap because they always appear together with other elements and cannot be separated easily or at sufficiently low cost.

The use of these rarer materials is increasing with the development of separation methods. A progress report on the use of the oxides of zirconium, thorium, cerium, and the rare earths would be quite extensive; much information, however, has been published on zirconia. Preston,[58] for example, has published a review on zirconia in glass. The application of cerium and the rare earths, however, has been less frequently discussed in the last two years although they certainly deserve the consideration of ceramists.

II. Zirconium Oxide

(1) Elemental Zirconium

Zirconium is by no means rare; in fact, zirconium silicate occurs in all quartz sands, but this occurrence is always in dilute form.

The element, zirconium, was discovered by Klaproth in 1789 and is known to occur in amorphous, crystalline, and graphitic forms. It may be drawn and polished, it has a specific gravity of 6.9, and it melts at 2330°C. Ductile zirconium leaves durable scratch marks on glass which have the appearance of silver.[28] This recent discovery could be used for decorating purposes.

(2) Raw Materials

The most important zirconium mineral is zircon or zirconium silicate, $ZrSiO_4$ ($ZrO_2 \cdot SiO_2$, 67.2 % of ZrO_2 and 32.8% of SiO_2). Commercial products[8] may contain 66% of ZrO_2 and 31% of SiO_2 with aluminum, titanium, phosphorus, iron, and water. Its melting temperature is 2200° to 2550°C., depending on its purity.[8] It is found in Australia, India, Brazil, and the United States. Zircon is usually concentrated and separated from the minerals occurring with it as follows: (1) The quartz is removed on a Wilfley-type wet concentrating table; (2) ilmenite and garnet are removed by a low-intensity magnetic separator; (3) monazite is separated on a high-intensity, cross-belt Wetherill separator; and (4) rutile is separated from zircon by electrostatic means or by flotation.

In the glass and enamel industries in which zirconium is employed in the presence of silica, zircon is a suitable source of zirconium oxide. Zircon resists the action of chemicals, especially acids (except hydrofluoric acid). With alumina, it forms a ternary eutectic at 1600°C.,[59] its hardness is 7 to 8 mohs, and it has a specific gravity of 4 to 4.7 and a mean refractive index of 1.95. Its coefficient of expansion,[16] which is 4.5×10^{-6}, is low in comparison to that of other crystalline materials (Cr_2O_3, 12 and mullite, 5.4). Its thermal conductivity of 0.004 to 0.007 gm. cal./cm.²/°C./sec. is also relatively low (fire clay 0.035 and Carbofrax, 0.036). When it is heated above 1600°C. and quenched, the zircon largely decomposes into zirconium dioxide and a glassy silicate phase.[7] The low-density zircons show this high content of free zirconium oxide. On reheating to 1450°C., ZrO_2 and SiO_2 recombine to form the silicate. In gemstones, the zircon with much free ZrO_2 is characterized by green colors.[12(b)] On reheating to 1450C°., the density and hardness increase[12(c,e)], and the color changes to blue or yellow. The low-density zircons contain less crystalline matter because part of the free ZrO_2 and SiO_2 is amorphous.

Zircon minerals were formerly classified in different groups according to their specific gravity. The different specific densities, however, do not correspond to distinct varieties but represent different grades of decomposition.

The approximate melting diagram of zircon has been determined by Washburn and Libman[81]; its expansion curve, when heated above 1800°C., necessarily depends on the cooling process.[33]

Baddeleyite (zirconium oxide, ZrO_2) was discovered in 1892 by Hussak in Brazil and by Fletcher in Ceylon. The Favas deposit,[71] first extensively described by Weiss and Lehmann,[83] contains about 85% of ZrO_2. Zirconium oxide also occurs in North America, Australia, and the Ural Mountains.[4,8] It melts at 2700° to 2900°C. Its purity is 80 to 94%, and it may be even as high as 98%; the impurities are chiefly SiO_2, Fe_2O_3, Al_2O_3, and TiO_2. For high temperature use, such as in refractories, iron and titanium oxide are objectionable and should be removed by acid treatment.

Zirconia is also a by-product of monazite. Historically,[4] the use of monazite for the preparation of cerium compounds in the gas-mantle industry first stimulated research on zirconia. Pure zirconia is prepared by electrofusion[24(e),70] or by leaching the finely ground ore with sulfuric acid and roasting the sulfate. The intermediate formation of carbides has been important for the production of ZrO_2 opacifiers in the enamel industry and will be discussed together with other properties of the carbides.

(3) Properties of Zirconium Oxide

The application of zirconium oxide in ceramics is justified because of the combination of its valuable properties, some of which are outstanding. Zirconium oxide has a high degree of hardness, 6 to 7 mohs[24(e),70] and excellent chemical resistivity. The coefficient of expansion of zirconium oxide[24(e),70] has often been given as low as 8×10^{-7}. Weiss[83] measured this low coefficient in 1909, using a specimen which would now be considered to be at least partly vitrified. Cohn[14(e)] found much higher values, based on the measurements of several hundred specimens. The values which he obtained for the coefficient of expansion varied between about 5×10^{-6} and 10×10^{-6}, depending on the thermal history. The inversion of zirconia to modifications of a smaller specific volume is responsible for the irregularity of the expansion-temperature curve. The expansion behavior, including the changes in volume due to inversion, rather than the coefficient of expansion of individual modifications, must be considered if material of this type is characterized for practical use.

Low expansion, high chemical resistivity, and hardness are indicative of the properties which make glassy silica the most important ceramic raw material. Zirconium oxide differs from silica glass by its extremely high melting point of 2900°C.,[21] which is considerably decreased by impurities but which is well above 2000°C. in all available materials.[4] The combination of these properties forms the foundation for its use as a refractory. Even when it replaces silica, zirconium oxide increases the chemical inactivity of glass, and zirconium-containing glasses have remarkable elastic properties. Zirconium oxide is neither a pronounced base nor a pronounced acid, and, therefore, it is not readily attacked by acids or by bases. It is inactive with respect to magnesium and beryllium oxides above 2000°C.[48] The inert character of zirconium oxide is important in its use as an opacifier in enamels. This application is based on its insolubility in many silicate glasses and, frequently, on the separation of the free zirconium oxide from the silicate by means of alkali. Zirconium opacifiers, consequently do not react with colored pigments, and this property makes them suitable for opaque colored glazes.

Zirconium oxide is attacked by fluorides and carbon[4]; it is attacked by carbon more strongly than is beryllia. Another characteristic property is its low heat conductivity. Zirconium oxide has a specific gravity[70] of 5.8 and a high refractive index[45,68] of about 2.3. The refractive index and other physical properties, especially the specific gravity, have been found to depend on the thermal history.

Several workers have determined three modifications of zirconium oxide[9,17,29,70] as follows: (1) a monoclinic low-temperature modification stable below about 1000°C., (2) a tetragonal modification, and (3) a cubic modification. Goldschmidt[29] and Tin Yung Ting* found that the cubic structure appeared to be more stable when magnesium oxide was present. Ruff and Ebert[67(a)] were able to verify in the purest materials only two modifications, a low-temperature monoclinic form, C, and a high-temperature tetragonal one, B. The inversion is reversible. The high-temperature tetragonal modification, B, may also be obtained at temperatures lower than 500°C. The metastable tetragonal form was investigated by Clark and Reynolds.[13] It changes to the stable monoclinic form above 600°C., and in the presence of silica, the metastable tetragonal form may prevail up to 1000°C.

Cohn [14(e)] reported that zirconium oxide, prepared by prolonged heating above 2000°C., occurs in two trigonal modifications, namely, A_1 and A_2. The material used in his experiments contained some magnesia. Cohn[14(b)] more recently prepared vitreous zirconium oxide. The crystalline material was fused in 30 seconds at above 3000°C. in a Straubel sun furnace. The glass is transparent, yellowish, hard, and highly acid resistive. This ZrO_2 glass is believed to be present in a ZrO_2 product of low refractive index prepared from the electrically fused oxycarbide. It probably was present in the material investigated by Weiss and Lehmann.[83]

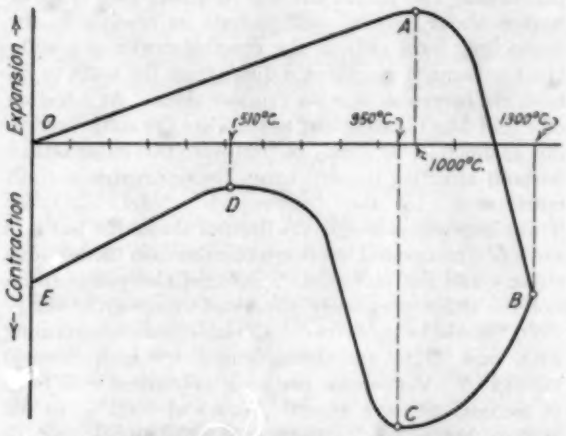

FIG. 2.—Expansion behavior of ZrO_2; (according to Cohn) monoclinic with some cubic material, fired at 1300°C.; *O*, heating started; *A*, modification change at 1000°C.; *B*, cooling starts, contraction still continues; *C*, 950°C. re-formation of monoclinic lattice; *D*, new contraction of 510°C. leading to shorter piece than original specimen.

The changes into modifications of different specific gravities is a real limitation of the use of purest zirconia for refractories. The high melting point, low expansion coefficient, and high resistivity are useful in refractories, but crucibles and brick[36] prepared from such material are liable to disintegrate.[62] The modification change also accounts for the irregular expansion behavior of zirconium oxide. Predominantly monoclinic material, prepared at 1300°C., ceases to expand around 1000°C.; after an interval of low expansion, it starts to contract because of the formation of the denser modification. When the material in this expansion test is cooled from 1300°C., it continues to contract because of the sluggish type of the inversion. Around 950°C., further cooling causes expansion as the more voluminous monoclinic modification is again formed. Below 500°C., the inversion stops even though some tetragonal ZrO_2 is still

* Cited by Ruff and Ebert, reference 67(*a*).

present. These tetragonal crystals are now metastable, and the mixture contracts with further decrease of temperature to a volume smaller than that of the original sample. Figure 2 illustrates this behavior and is only one example of the detailed description of various preparations by Cohn.[14(a)]

III. System MgO–ZrO_2

Inasmuch as the zirconia inversion is inhibited by magnesia, the system MgO–ZrO_2 forms an important basis for the understanding of the behavior of high-temperature refractories (see Fig. 3). The phase relationship was established by Ebert and Cohn.[23] In order to stabilize the cubic ZrO_2 lattice,[67(a)] an addition of 4% of magnesia is sufficient. Even the monoclinic zirconia changes to the cubic form when it is heated above 1700°C. with 4% of MgO. On cooling, the cubic form remains stable, which means that no volume change will occur with cooling, reheating, and repeated thermal changes.

The action of MgO may be demonstrated by an experiment. The purest zirconia in a magnesia crucible heated above 1700°C. will change on cooling to the monoclinic form only in the crucible center if a sufficient amount of magnesia diffuses from the walls to inhibit the inversion near the crucible sides. An addition of 4% of MgO is sufficient to stabilize the cubic lattice, but as much as 40% may be built into this cubic lattice without affecting its structure; the approximate limit corresponds to the "compound" $2MgO{\cdot}3ZrO_2$.[67(a)] This composition is only the limit of the series, but it is not a real compound because a maximum in the liquidus curve could not be found.[23] Neither this composition nor the other previously suggested compound, $MgO{\cdot}ZrO_2$,[80] could be confirmed. Ceramic bodies containing ZrO_2 and MgO are characterized by high flexural strength.[62] Very dense bodies are obtained with 90% of zirconia where a eutectic occurs at 1600°C. in the system MgO–ZrO_2.[62] Higher MgO contents lead to shrinkage of more than 20%.[62] The system has recently been studied by Zhirnova[85] in Russia in connection with practical applications.

IV. System TiO_2–ZrO_2

The system TiO_2–ZrO_2 is important because ceramic bodies with unusual electrical properties may be obtained by the addition of titania. The characteristic properties of rutile (TiO_2) are (*a*) a high mean value of the dielectric constant of about 80 at 3×10^8 cycles per second, (*b*) a decrease of the dielectric constant with increased temperature in rutile bodies (the dielectric constant usually increases with temperature), and (*c*) a low power loss at high frequencies.

The system TiO_2–ZrO_2 shows no compounds. It is of the simple eutectic type. The eutectic temperature is 1750°C. for a composition of 20% of ZrO_2. When less than 20% of ZrO_2 is used, the increase of the melting temperature is slight; this allows the production of very dense bodies in this area so that bodies of high chemical resistivity can be obtained. The firing shrinkage decreases between 95% and 56% of TiO_2 but increases with a further increase of ZrO_2. Bodies with a higher content of ZrO_2 (75% or more) disintegrate because of the inversion of the tetragonal to the monoclinic modifications of zirconia. Because no compounds occur in the system TiO_2–ZrO_2, the dielectric constant is almost proportional to the amount of titania. Zirconia may be built into the rutile lattice in amounts of 10 to 16% without structural change. This limit is important for the preparation of dense materials. Whenever a body has been found to meet the requirements, X rays have revealed that zirconium oxide had been built into the TiO_2 lattice.[10] The specific weight of such bodies is higher than that calculated from the compositions; this fact confirms these findings.

Bodies made with the composition of this mixed crystal area show lower dielectric losses at low frequencies than does pure rutile. The high losses at low frequencies in rutile are probably due to the reduction of TiO_2 to semiconducting lower oxides which, according to Meyer* still contain more oxygen than Ti_2O_3 and which have the bluish color often observed. According to Albers-Schoenberg,* the ZrO_2 in the TiO_2 lattice is a protection against these oxygen losses and the formation of other conducting C- or N-compounds.[10] The color of titanium-zirconia bodies, on the whole, is brighter than that of pure rutile bodies.

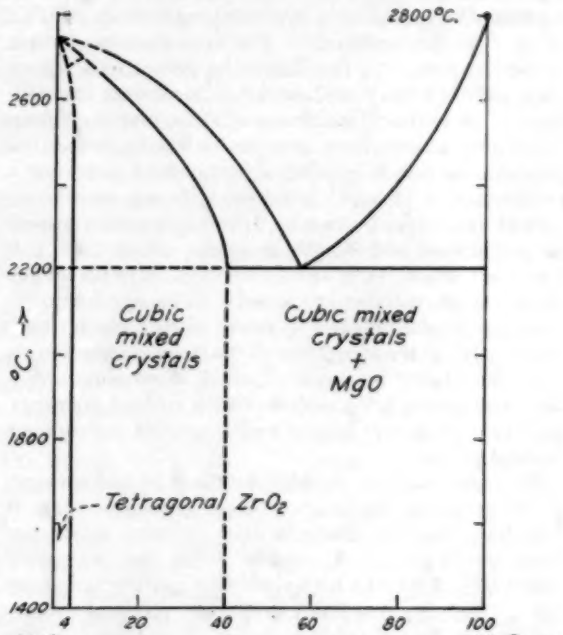

FIG. 3.—The system ZrO_2–MgO (according to Ebert and Cohn).

V. Ternary System MgO–ZrO_2–TiO_2

The work of Rieke and Ungewiss[87] contains valuable and detailed information of specific ceramic properties of various ternary compositions.

Of the three components, TiO_2 is the strongest acid and MgO is the strongest base. Zirconia is neither a

* Cited by Büssem, reference 10.

strong acid nor a strong base; MgO, therefore, tends first to react with TiO_2 to form Mg_2TiO_4 ($2MgO \cdot TiO_2$). Only an excess of magnesia over this ratio is believed to react with ZrO_2 to form $MgZrO_3$ ($MgO \cdot ZrO_2$), but even in the case of an excess of ZrO_2 over "compounds" like $MgZrO_3$, no free ZrO_2 may be detected in X-ray pictures. There is no doubt that ZrO_2 might be built into the lattice of Mg_2TiO_4 just as it can be built into the TiO_2 lattice. A comparison of the ternary bodies to those containing only MgO and ZrO_2 or TiO_2 and ZrO_2 shows the improved impact strength of certain compositions and the high flexural strength of compositions high in MgO content and containing some ZrO_2. Rieke and Ungewiss[62] attribute this latter improvement to the "compound" $MgO \cdot ZrO_2$ and to free MgO. The translucency is increased by Mg_2TiO_4. The lowest expansion is caused by increased ZrO_2; the highest, by an increased content of MgO. The three component bodies possess higher dielectric losses than the binary bodies of the system ZrO_2–TiO_2 because the structure is less homogeneous. The dielectric loss is usually increased if more phases appear.

VI. Other Systems Containing ZrO_2

Inasmuch as 4 molar % of magnesia prevents the inversion of zirconia, this result may be applied to the production of stable zirconia refractories. The question may arise as to whether MgO is unique in achieving this effect.[67(a)] Only refractory oxides, however, can be considered. Beryllia, alumina, and zinc oxide are not capable of this effect because they are hexagonal. The elements forming refractory oxides which fit into the cubic lattice of stabilized zirconia are listed, given in the order of the radius of their ions as follows:

Mg	0.78	Y	1.06
Sc	0.83	Th	1.1
Zr	0.93	La	1.22
Ce	1.02	Sr	1.27
Ca	1.06	Ba	1.43

The ionic radius of scandium is closest to that of magnesium. Scandium oxide actually does stabilize the cubic zirconia lattice just as well as magnesia. Some favorable influence in a dilated cubic lattice is exerted by CeO_2, CaO, and yttria. Crucibles with 3% of yttria have been found to be good,[67(c)] and lime binders are used to advantage.[16] Refractories with 25 to 50 % of ZrO_2 and 40 to 50% of CaC, with and without chromite, were pressed by Pole.[57] The oxides of elements with larger ionic radii fail to stabilize the cubic lattice of zirconia. Iron oxide would meet the crystallographic requirements (ionic radius about 0.83), but as it lowers the melting point too much, it cannot be used for zirconia refractories.

The systems of ZrO_2 with silica and Na_2O are important in the production of enamels and glazes and will be briefly discussed with the zirconium opacifiers. Zirconia occurs in talc bodies in which zirconia may be used to lower the expansion.[76]

VII. Other Compounds

Of the other compounds of zirconium, the carbides have found some use in abrasives and as an intermediate product for the preparation of the pure oxide. Zirconium carbide was first synthesized by Troost[77] in 1893, who reduced zircon with sugar coal in an electric furnace. The method was improved by Renaux,[61] who used calcium carbide in the *status nascendi* and who followed the classical study on carbides of Moissan.[60] Wedekind[82] used the carbide for the preparation of the pure oxide, with the chloride as an intermediate product. In 1908, Lehmann* prepared the oxide by igniting, in an oxidizing atmosphere, a carbide made in the presence of lime. One modern method of manufacturing zirconium oxide opacifiers by Kinzie and Hake[42(b)] is based on these chemical reactions. The carbides have the high melting points (about 3000°C.), which are characteristic of good abrasives.

Zirconium carbides scratch quartz,[4] and they are used for cutting materials. A superrefractory zirconium carbide with a melting point of 3800°C. is mentioned by Charrin.[11]

Zirconia with silicon carbide compositions† has been used in refractories, zinc muffles, and flameless surface combustion.[4]

The boride of zirconium has a hardness coefficient and high fusing temperature similar to the carbides and borides of some other elements which were studied by McKenna.[47]

VIII. Whiteware and Refractories

The foregoing discussion has indicated that zirconia is a valuable constituent of special porcelain for chemical and electrical purposes because of its chemical resistivity, its low thermal expansion, and its strengthening action,[78] especially in ZrO_2-TiO_2 bodies of high dielectric constant and of low dielectric loss. Bodies have been made containing as much as 70% of zircon; the thermal-shock resistance of these bodies is exceptional because of the combined effect of elasticity, strength, and low expansion.[8] Talc bodies of low expansion may be produced[76] by the addition of ZrO_2 to cordierite. If a given volume of flint is to be replaced by ZrO_2, the high specific weight of the latter must be considered.

McMahon[48] has reported that when zircon is added to porous talc bodies the temperature range increases between shivering and crazing. It is a characteristic of talc bodies that firing the body a little too high causes crazing and firing the body a little too low causes shivering because high-fired bodies have a lower coefficient of expansion than have low-fired bodies. In one case, the complete substitution of zircon for talc reversed the expansion characteristics and a body was formed which had a higher expansion when it was fired at a higher temperature. A partial substitution leads to a beneficial compensation which is responsible for the increased firing range.

The background for the use of zirconium oxide in refractories has been discussed. The high melting point,

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* Cited by Bayer, reference 7.

† Brit. Pat. 4080 (1915); U. S. Pat. 1,159,624 (1915); Ger. Pat. 287,122 (1913); and to Knöfler, Ger. Pat. 284,395 (1914).

the low expansion, the stability of the crystalline form in the presence of small amounts of certain oxides like magnesia, and the inertness to many chemicals and metals are the important properties which have stimulated research.

J. A. Harker proposed the use of zirconium oxide as a refractory about 1900, and the National Physical Laboratory of the University of Sheffield, England, prepared some brick using organic binders. In 1904, Pyfahl obtained a patent on zirconia muffles and tubes.[4] In 1906, the Heraeus Company in Germany produced zirconium oxide crucibles for melting quartz,[25] and crucibles were made from zircon about 1910.[5] The owners of the South American ores had just sent large collections to Munich, where Weiss and his collaborators[52] started their extensive research work. In cooperation with C. W. Goebel and Sons, Grossalmerode, crucibles containing MgO and ZrO_2 with P_2O_5 as a binder were developed in 1909. The Nernst Lamp Company produced crucibles which contained an alkaline flux. In an independent investigation, Bayer[7] prepared the first zirconia crucibles with a binder of zirconium hydroxide. A large-scale experiment was made at this time in the Remscheid Steel Works in Germany.[4] The management of this plant decided to take the chance of using zirconium oxide in a Siemens-Martin furnace and reported that in spite of the price considerable savings could be made. Ruff and Lauschke[67(b)] at that time pressed moistened powders into crucibles; they had already discovered that small additions of other oxides prevented disintegration. Podszus[56] heated zirconium oxide to 2400°C. and prepared from almost colloidally dispersed material very stable zirconium oxide ware without any binder. From this period on, work with zirconium oxide refractories never ceased. In England in 1916, Audley[4] drew the attention of the ceramists to the possibilities of zirconium oxide. European interests in 1915, foreseeing the importance of Zr ores, tried to monopolize the South American production.[49(a)]

Important work on zirconium-containing refractories has been carried on in various laboratories in the United States. Between 1916 and 1920, original research was carried out in the research laboratories of the Foote Mineral Company by Meyer[40] and other workers.[26] Granger,[30] in addition to new contributions, made the American ceramist familiar with all of the previous work. At the end of the first World War, zirconium oxide refractories appeared as a commercial article (Zirkite, Zirconalba). After five years of concentrated research on zircon and zirconium oxide, Comstock[16] published his findings in the various fields of refractories in 1933. Zircon brick, lightweight brick, steel-ladle nozzles, open-hearth refractories, magnesite-zircon brick, combinations with carbides, fused alumina, fire-clay, and zirconium oxide crucibles were studied. Examples of other recent contributions in this field are the studies of Swanger and Caldwell,[73(b)] Heindl,[33] Thompson and Mallet,[71] Roller, Rittenberg, and Gabriel,[66] as well as those of Partridge and Lait[54] in England. The most typical application of zirconium-containing refractories is in the melting of noble metals, such as platinum, rhodium, and iridium. When electrically sintered zirconia was commercially available, Jordan, Peterson, and Phelps[39] found that the material could be recommended for this purpose and that magnesia was still preferred for melting iron and nickel. The detailed description of the methods in making these zirconia-containing materials and their use is available in the papers covering the work of these investigators.

Magnesite is still generally preferable as a refractory in contact with basic slags in the open-hearth process. This does not mean, however, that the consideration of zirconium does not enter the discussion of the problem of an ideal refractory for this purpose. Zircon, the silicate of zirconium, at the present time seems to be the only source that approaches the price requirements, but it is not resistive enough toward basic slags. Magnesia-zircon brick, as suggested by Kraner,[16] are slightly inferior to magnesite brick with regard to slag attack, but they are superior in high-temperature strength and spalling resistance.[16,60] The continued shrinkage on heating the fired brick is much lower with a composition of about 65% of MgO and 35% of ZrO_2 than with either of the pure oxides.[60] Zirconium oxide is not attacked by iron silicate and basic slags so much as is zircon.[36] Free silica, however, in zirconium oxide combines more easily with the bases of the slag than the silicate,[30] and a pure zirconium oxide is still excluded because of its high cost.

Because with the use of a highly refractory material such as zirconium oxide there still exists the problem of obtaining density, the use of binders is discussed in section IX. The use of zircon for steel-ladle nozzles has been found promising.[16] The limitations which are based on the attack of basic slags do not prevail in the case of glass, which is more acidic. The introduction of ZrO_2 to glasshouse refractories is promising from the chemical viewpoint, and the difficulties involved are merely economical and technological.

Zircon with alumina was found to be somewhat superior to magnesite in contact with copper but not enough to justify the higher expense at the time of the investigation.[16] The glazing of zirconia refractory ware with a zirconium glaze was tried by Arnold[3] in 1918 without success.

IX. Binders and Cements

Zirconium refractories have been pressed and tamped without any binding material. In many cases, binding materials were used which are common in the technique of plastic mixtures, such as clay, phosphates, chlorides, lime, sodium silicate, and caustic soda.[16,67(c,b)] It is often desirable to avoid inorganic binders which act as fluxes and to use organic binders such as goulac or dextrin.[67(c)]

The essential feature in making superrefractories out of rare oxides seems to be that the oxide, for example, ThO_2 or ZrO_2, is shaped without the help of plasticizing materials such as clay. Only in this case can full advantage be taken of the high melting point. In order to plasticize ThO_2 or ZrO_2, two methods may be used, namely, (1) the addition of a salt such as $ZrCl_4$, which

readily hydrolyzes and therefore covers each grain with a film of adhesive hydroxide,[67(c),73(b)] and (2) the treatment of the highly fired oxide with acids and bases alternately so that a layer of hydroxide is formed around each grain. These two methods permit the use of oxides which have been fired at high temperatures so that no excessive firing cracks occur. The grain-size distribution is particularly important.[67(c)] An optimum proportion may be obtained only if the distribution of the grain sizes allows the densest packing. Experiments with hydrated zirconium oxide binders were started as early as 1909.[6]

High-refractory cements containing ZrO_2 have been prepared with sodium silicate and zircon, and cements for high temperatures have been made up with NaCl, goulac, and dextrin.[14(e)] Many formulas are based on a mixture of zircon sand with finely ground zircon or alumina in the ratio 2 to 1 with various binders used.

X. Glass

The introduction of zirconium oxide in glasses seems to have been limited chiefly by price considerations although the decreasing cost of zirconium-containing materials has partly removed this obstacle. It is not known to what extent these changing conditions have encouraged the glassmaker to take advantage of the valuable properties of this glass constituent in the past few years. Several years ago, the use of ZrO_2 in the glass industry was comparatively small. Nothing has been reported concerning the use of ZrO_2 in glass since these conditions were discussed by Preston[58] in 1938. In some experimental glasses, CaO was replaced by ZrO_2 as early as 1909.[53] Systematic work on zirconia in glasses has been conducted in England[19,20] and in the United States.[35] The essential influence of zirconia on glasses is the improvement of chemical resistivity and elasticity. The modulus of elasticity is lowered by 60%; the modulus of rigidity is also noticeably lowered[35] if 4% of ZrO_2 is substituted for lime. The combination of favorable elastic properties with a fairly low coefficient of expansion makes these glasses extremely resistant to thermal shock. Of particular interest is the use of 1% of ZrO_2 in a pure silica glass according to the Wolf-Burkhardt invention. This glass, Siloxide Z, was prepared about 1910 and described by Thomas.[74]

Because zirconia has been used in enamels more frequently than in glass, it is usually considered to be an opacifier which is insoluble in the melt. At the temperature and long melting times used in the glass industry, however, up to 20% of ZrO_2 may easily be dissolved. The improvement of chemical resistivity and elasticity is accompanied by a rise in melting temperature, viscosity, and annealing temperature. The temperature coefficient of viscosity decreases and the glass becomes "shorter."

Some of the improvements in the chemical resistivity of soda-lime-silica glasses by ZrO_2, which were demonstrated by the work of Turner and his collaborators,[19,20] could not be obtained with other constituents. These investigators found (1) that zirconium oxide is the only glass constituent which, by replacing alkali, increases the resistivity toward water, acid (HCl), NaOH, and Na_2CO_3; (2) zirconium oxide, used to replace alkali, increases the resistivity toward these agents more than any of the other oxides investigated, namely, Al_2O_3, TiO_2, ZnO, MgO, PbO, CaO, and BaO; (3) the resistance of ZrO_2-containing glasses toward boiling alkali is outstanding (Al_2O_3 in this case is not so efficient); when more basic oxides are introduced, glasses generally are attacked more readily by NaOH than by Na_2CO_3; the effect of ZrO_2 is intermediate in this respect; and (4) increased resistivity toward H_2O, HCl, and NaOH is observed even if silica is partly replaced by zirconium oxide. Glasses of this type are being used in gauge tubes for boilers.[58]

Long[72] replaced with ZrO_2 a part of the silica in high lead oxide glasses, which are used for protection against short-wave rays, to improve the working properties.

XI. Enamels and Glazes

The use of zirconium compounds in enamels and glazes has the widest application in the ceramic field and is increasing steadily. If the zirconium compound is dissolved in the vitreous component of the enamel or glaze, it improves the resistivity toward chemical attack and the elastic properties in the same way as it does in glasses. Zircon has a beneficial influence on the resistance to crazing in semivitreous dinnerware glazes,[34] and it increases the resistance to abrasion as determined by the gloss retained after a test procedure proposed by the Porcelain Enamel Institute.[15(b)]

The chief application of zirconium compounds to enamels and glazes, however, is as an opacifying agent. The difference of refractive indices between the opacifier and the enamel is one determining factor for opacity. It does not matter whether the refractive index of the opacifier is high or low in comparison to that of the enamel, which is about 1.5. Tin oxide, with a refractive index of 2.0, was generally used in the 19th century. In the search for new opacifiers at the beginning of this century, attention was focused on materials with a refractive index higher than 2.0; that of zirconium oxide is 2.4. From this relation and from the specific gravities of both oxides (ZrO_2 5.8 and SnO_2 6.95), the effect of one weight unit of zirconium oxide may be calculated as 2.8 times higher than that of tin oxide.[45] This is demonstrated in organic liquids whose refractive index is that of enamels. This relation, however, holds only for moderate concentrations of the opacifier.

Materials containing zirconium became more generally available when this element was used in the gas-mantle industry near the beginning of this century. The use of pure zirconium oxide was tried unsuccessfully by the Guestrow Company in 1906.[31,37]

At about this time, Kreidl[43] fused the zircon ore with alkali and removed a part of the alkali necessary for the decomposition of the silicate by leaching with water or weak acids. An opacifier was obtained, called "Terrar," which found general use in Europe until 1930. The gas opacifiers from that time gradually replaced a part of the metal oxide opacifiers in Europe. This first application of zirconium compounds as enamel opacifiers was based on the fundamental discovery that

the control of the alkali used in the preparation from the silicate is decisive. The alkali remaining in the opacifier and the thermal history of the opacifier was found to be more important than its purity. The disintegration of the zirconium silicate took place at temperatures below 600°C.[43(e)] Only a part of the alkali, however, was removed; the remaining alkali concentration was approximately 10%.[43(a,b,c)] Other factors controlling opacification were (*a*) the presence of salts of weak acids, such as those salts formed by the reaction of the leaching acids and sometimes deliberately left behind,[43(g)] (*b*) the relation of water of hydration to alkali concentration,[43(f)] and (*c*) the control of grain size.[43(h)] Zirconium silicate treated according to these processes was calcined to form the opacifier. Similar products were obtained from zirconium oxide and silica.[43(a,b)]

The manufacture of zirconium opacifiers in the United States increased considerably around 1915. Many suggestions were advanced by Kinzie and his collaborators,[42] especially in the field of opacifiers made from the silicate, which was heated with carbonaceous material to form oxycarbides and which was calcined with oxygen to form the pure oxide.[42(f)] The earlier work on the formation of zirconium carbides and the preparation of pure zirconium oxide from the carbide is given in the discussion of the carbides in section VII. Various opacifiers were derived from the electrofused type,[42(e)] and these could be improved by heating with about 10% of alkali at elevated temperatures.[42(d)] This action verifies the fundamental influence of alkali on the stability of the opacifying phase which was the basis of the Terrar process. Other zirconium compounds, such as the double silicates of different bases, were also investigated.[42(b,c)]

The reduction of zirconium silicate by carbonaceous material takes place at temperatures above 2000°C. The larger part of the silica volatilizes, but a small part remains as silicon carbide and should be removed by reduction. A cyanonitride is formed when the Barton[5] process is used, and oxycarbides are formed in a later process of Kinzie and Hake.[42(h)] The final oxidized product has a low refractive index, which is believed to be due to the presence of a vitreous phase of ZrO_2 containing small ZrO_2 crystals. In the opacifier, crystalline material and any material which recrystallizes is efficient. The presence of a ZrO_2 glass in one of the electrofused ZrO_2 opacifiers probably influences the solubility of the opacifier in the enamel.

At the present time, the use of zirconium opacifiers may be considered to be established in ceramics. In the process for their preparation, they may be divided into three types,[69] namely, (1) those prepared from zircon ore which is heated with alkali according to Kreidl[43] (Terrar), (2) those obtained from electrofusing zircon with carbon and oxidizing the intermediate products,[5,41] and (3) those precipitated from salt solutions by alkali.

A distinction is made between mill and frit opacifiers according to their use. As mill opacifiers, they are obviously applicable to glass enamels even if they are somewhat soluble, owing to the short reaction time. The development of zirconium opacifiers, therefore, was limited at first to their use as mill additions; the frit opacification was studied later and was used in enamels in Europe shortly after 1915.

With respect to the history of the solid phase, (*a*) zirconium oxide may be insoluble in the enamel frit or (*b*) it may reprecipitate from its solution.[41(a,b)] A part will always be soluble and contribute to the properties of the glassy phase.

The zirconium opacifiers are distinguished by their chemical stability as compared with other opacifiers of high refractive index. This stability permits their application in combination with colors which would react with other opacifiers.[21]

XII. System SiO_2–Na_2O–ZrO_2

The equilibrium conditions in the ternary diagram SiO_2–ZrO_2–Na_2O were determined by d'Ans and Löffler,[3] whose findings show that the development of an opacifier containing these three components is necessarily complicated. The binary compound, $ZrO_2 \cdot SiO_2$ ($ZrSiO_4$), may be obtained at different specific gravities, but this behavior as previously stated, is no longer attributed to the existence of modifications. Three ternary compounds, $Na_2O \cdot ZrO_2 \cdot SiO_2$, $2Na_2O \cdot 2ZrO_2 \cdot 3SiO_2$, and $Na_2O \cdot ZrO_2 \cdot 2SiO_2$, were established. Some properties of the first two are shown in Table I.

TABLE I

PROPERTIES OF TWO TERNARY COMPOUNDS

	Composition	
	$Na_2O \cdot ZrO_2 \cdot SiO_2$	$2Na_2O \cdot 2ZrO_2 \cdot 3SiO_2$
Formula	Na_2ZrSiO_5	$Na_4Zr_2Si_3O_{12}$
Melting point (°C.)	1477 (incongruent)	1540 (congruent)
Specific gravity	3.6	2.88
Appearance	Plates	Rhombohedra
Optical character	Biaxial	Uniaxial

Because of the incongruent melting point of Na_2ZrSiO_5, mixtures of a composition of $Na_2O \cdot ZrO_2 \cdot SiO_2$ will yield different products when they are subjected to different heat-treatments. The extreme cases are (*a*) a homogeneous crystalline phase of the zirconium silicate of sodium and (*b*) free ZrO_2 in a glassy matrix.

The possibility of obtaining free ZrO_2 and glass under certain conditions explains the success of the alkali treatment of zircon in the manufacture of opacifiers. This reaction may be explained by the fact that Na_2O is the strongest base of the three oxides and SiO_2 is the strongest acid. These two oxides will react before their reaction with the amphoteric ZrO_2 after Na_2ZrSiO_5 decomposes into $Na_2O + ZrO_2 + SiO_2$ at high temperatures. A similar effect may also be obtained by boric fluxes in the presence of which the zirconium silicates decompose.[53]

XIII. Stability and Solubility of Zirconium Oxide in Enamel Frits and Glazes

The opacification of the enamel frit depends not only on the relatively high refractive index of the opacifier but also on the stability and solubility of the opacifying phase. In the case of a zirconium opacifier, it is the

zirconium oxide which has to be stable, perhaps chiefly in the tetragonal form.[32] A comparison of the refractive indices of some possible zirconium compounds illustrates this fact, if it is remembered that the refractive index of the frit is about 1.5.

ZrO_2	2.4
$ZrSiO_4(ZrO_2 \cdot SiO_2)$	1.95
$Na_2ZrSiO_4(Na_2O \cdot ZrO_2 \cdot SiO_2)$	1.72
$Na_2ZrSi_2O_7(Na_2O \cdot ZrO_2 \cdot 2SiO_2)$	1.65
$Na_2ZrO_3(Na_2O \cdot ZrO_2)$	1.9
$Na_4Zr_2Si_3O_{12}(2Na_2O \cdot 2ZrO_2 \cdot 3SiO_2)$	1.69

Only the lead zirconium silicate has a higher refractive index, but it is yellowish and stable only in lead glasses, which also have a high refractive index. The net difference, therefore, in refractive index between the glass and the opacifier would be small and, consequently, the opacity would be slight.

In glazes with a higher firing temperature, zirconium silicate might be more efficient than zirconium oxide in spite of the lower refractive index, according to Schulz.[69] He states that the lower solubility and the smaller gas content are important factors at a high firing range and that these factors compensate for the smaller opacifying power.

The stability of the zirconium oxide, however, may generally be assumed to be an essential condition of high opacification, that is, the ZrO_2 phase rather than any zirconates or zirconium silicates should be present in the undissolved or recrystallized phases. Alumina has been found to be necessary not only to lower the solubility of ZrO_2 in the glass but also to stabilize the ZrO_2 by preventing it from entering compounds. For several ternary fields of glasses, the limits within which ZrO_2 is stable as the undissolved or recrystallized phase have been determined by Löffler[45] as follows:

$K_2O \cdot Al_2O_3 \cdot 2\text{–}6SiO_2$	$ZnO \cdot Al_2O_3 \cdot 2SiO_2$
$BaO \cdot Al_2O_3 \cdot 2\text{–}6SiO_2$	$PbO \cdot Al_2O_3 \cdot 2SiO_2$
$Na_2O \cdot Al_2O_3 \cdot 2\text{–}4SiO_2$	$MgO \cdot Al_2O_3 \cdot 1.5SiO_2$
$CaO \cdot Al_2O_3 \cdot 2\text{–}4SiO_2$	

Because aluminum oxide is necessary, this oxide was kept throughout the study, and the limits of silica variations with different basic oxides are listed within which ZrO_2 is stable. Some alumina, however, may be omitted in the systems having a large range of SiO_2 variations, such as $BaO \cdot Al_2O_3 \cdot 2\text{–}6SiO_2$. If silica were to be increased over the limit, ZrO_2 would combine in compounds of lower refractive index, for example, $ZrSiO_4$. It is dangerous to decrease the alumina if the basic oxide present tends to form double silicates with zirconium as in the case of K_2O. With zinc oxide, however, no zinc zirconium silicate is formed, and some of the alumina, therefore, may be omitted. These conditions partly explain the known fact that zinc and aluminum oxides are beneficial; the influence of these oxides on the solubility of ZrO_2 is also pertinent.

Considering the practical applications in the enamel industry, it is significant that Löffler found an increase of the stability of ZrO_2 even in the presence of more silica whenever the opacifier had been introduced in combination with alkali. The effect of an opacifier of compositions related to $Na_2O \cdot ZrO_2 \cdot SiO_2$ is the creation of a local alkaline zone around the ZrO_2 which acts as a protection against its reaction with the siliceous melt.

Another protection has been successfully tried in glazes of the tile industry. Ground zirconium oxide-containing frits may be used, the composition of which had been established according to the preceding fields of stability. Many commercial frits may be represented as combinations of these fields.

The stability of zirconium oxide[45] does not seem to be influenced by cryolite, B_2O_3, and P_2O_5, but ZrO_2 tends to stabilize fluorine in the secondary fluorine opacification.[16(e)]

The dependence of the solubility of ZrO_2 on the composition of the base frit was observed when experiments with frit opacification were first carried out; no detailed reports, however, had been published in Europe. The principal rules governing the solubility of zirconium opacifiers in frits and glazes, according to the work of Kinzie and Commons,[42(g)] may be summarized as follows:

Alkali: Up to 15%, no influence.
Alumina: If less than 6%, considerable increase of solubility of ZrO_2; about 10% usually necessary; over 14%, excessive crystallization.
Zinc Oxide: Decreases solubility next to Al_2O_3; usually 5%.
Barium Oxide: Usually 5%; size and amount of precipitation increased; used chiefly as flux.
Calcium Oxide: 5%; increased use as flux.
Boron Oxide: Flux; increases solubility of ZrO_2.

King and Andrews[41(e)] reported on a series of experiments which demonstrate the influence of various oxides on the solubility of ZrO_2 in a sodium borate glass. The solubility is again decreased by Al_2O_3 and ZnO and also by MgO. If the Al_2O_3 content was low, sodium oxide and even the alkaline earths, seemed to increase the solubility. Of the fluorides, only cryolite had a specific effect. These authors also studied the interesting correlation of viscosity and solubility of (*a*) undissolved ZrO_2 and (*b*) reprecipitated ZrO_2 as the opacifying phase.[41(b)]

About 2% of ZrO_2 is always soluble in a frit[1] and contributes to the resistivity, elasticity, and brilliance of the enamel. The omission of alumina and zinc oxide may increase the solubility to 17%. Zirconium opacifiers are used in Europe at the present time chiefly in the powder process because zinc oxide is not a common constituent of the wet enameling frits.

In 1927, I. Kreidl and the Degea Company used glazes which contained zirconium, lead, and sodium chloride. At that time, experimental glazes in Germany contained 8 to 10% of ZrO_2 instead of 10 to 11% of SnO_2.[45]

The action of different zirconium silicates as opacifiers in white opaque American glazes is described by Commons[16(e)] (see Table II).

The zirconium oxide is not used ordinarily as a constituent of colored pigments because of the inertness which makes it so valuable as a white opacifier.[32] Robertson mentions that a yellow pigment, which con-

TABLE II

PROPERTIES OF DIFFERENT ZIRCONIUM SILICATES (COMMONS[11(b)])

Zirconium silicate	Gloss	Whiteness (color hue)	Opacity	Crystallization
Calcium	+	+	+	
Zinc	+	+	+	Soft
Lead	+		Little less	Some, due to softness
Magnesium		++ (more than calcium)	+ (as calcium)	If used alone, crystallization of other components takes place
Barium			+ (in small amounts)	Devitrifies
Zirconium spinel	+		+ (more than Zircopax)	

tains zirconium oxide and vanadium oxide, is stable at very high temperatures.[43]

XIV. Thorium Oxide

The use of thorium oxide in ceramics is limited. It has been used successfully, however, for laboratory crucibles, especially for the preparation of the purest platinum and for high refractory tubing for scientific apparatus.[73(b)] The preparation of high-grade platinum is important in making thermocouples. Neville[52] and Richardson* probably used unfused thorium oxide for making crucibles in 1923. Thorium oxide crucibles have been used by other investigators for similar work, such as the determination of the properties of pure rhodium.[52,65,73(c),75,84]

Thorium occurs in monazite and thorite.[24(b)] Deposits of monazite are found in the Carolinas, Texas, Brazil, India, Ceylon, the Ural Mountains, and Scandinavia. Thorium, which is not a rare element, composes 0.002% of the earth's crust. It is not highly concentrated, however, and its separation from the rare earths is tedious. After electromagnetic purification, the monazite is treated with sulfuric acid, and the rare earth elements are precipitated with oxalic acid. Thoria is white when pure, but when it contains traces of iron and of the rare earths it is brown.

Thorite is a hydrated silicate from which ThO_2 may be extracted with HCl and, after the removal of other metals with H_2S, the residue may be changed into sulfates and recrystallized as the double sulfate of potassium and thorium.

Thorium oxide is characterized by a high melting temperature, at least 2500°C.,[24(b)] by its basic nature, by some plasticity, and by its resistivity to acids when it is well calcined. Refractories made from fused thorium oxide are resistant to basic slags but not to silica. They resist temperatures close to 3000°C., and they are very heavy (specific gravity, 9.5 to 10); their thermal coefficient is low[52] as is their expansion. Thorium oxide refractories, which are reduced by carbon at high temperature to about the same degree as those made from zirconium oxide, are inferior in this respect to refractories made from beryllium oxide.[68] Thorium oxide is radioactive and, therefore, some conduction may be expected.

In the preparation of crucibles for Pt, c.p. thorium nitrate is reprecipitated as the pure oxalate and ignited at 1000°C. The crucibles are tamped into fused alumina crucibles either as dry powders or with petroleum ether; water, however, is never used.

Neville[52] had difficulty with cracking of ThO_2 crucibles. This is avoided by the use of fused ThO_2, which is a pinkish glass and was introduced in 1925 by Fairchild and Peters.[25] Swanger and Caldwell[73(b)] used this material successfully to melt potassium and iridium in crucibles tamped with a $ThCl_4$ binder into graphite molds. The use of such a binder is analogous to the zirconium salt binders described for zirconia.[67(c), 73(b)] The mixture is heated in the mold at 1100° to 1200°C. The firing to higher temperatures takes place after the removal of the mold because ThO_2 reacts with carbon just as zirconia does. Thorium oxide with a $ThCl_4$ binder has also been used for pressed crucibles. Tubes, which were made from slips containing more $ThCl_4$, were not preheated. Crucibles made from thorium oxide were also investigated in other laboratories.[54, 68] They are especially suitable for work in a vacuum because the vapor pressure of ThO_2 is very low.[68] Dense, impervious bodies for spark-plug insulators were made of sintered mixtures of Al_2O_3 and 6 to 50% of ThO_2.[46]

In glasses, the rare earths and thorium in combination with boron provide relatively low dispersion and high refractive index. The "normal" optical glasses show rising dispersion with increased refractive index when the composition is changed from crown to flint glass. These new glasses were discovered by Morey[51] and were modified by Eberlin.[22] A glass composition with high ThO_2 content is as follows: LaO 26.7, TaO 26.7, ThO_2 26.7, and B_2O_3 19.9%.

When conductors are sealed through silica glass, a glass as viscous as silica glass but of intermediate expansion is needed. For this purpose, glasses of high silica content (75 to 95%), containing more than 3% of ThO_2, of BeO, or of CeO_2 and combined with lime and zirconia,[55(b)] were suggested in England.[55(a)]

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RARE EARTHS*

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Abstract

Cerium oxide in glasses is the strongest oxidizing agent with respect to iron. In combination with neodymium oxide, which compensates the remaining hue, it represents the ideal decolorizer. Cerium oxide absorbs in the ultraviolet without affecting visible transmission. It is used as an enamel opacifier in France. Various other applications of the rare earths are listed, including those which are based on the abrupt division of the visible spectrum by the sharp absorption band of neodymium in the green.

I. Introduction

In the first paper of this series,† it has been shown that diluted occurrence, difficult separation, and economical factors rather than rarity cause the less frequent use of some special raw materials in the ceramic industry. This is true even in the case of the "rare" earths. They are found in the United States, Brazil, India, Australia, and Scandinavia. The main reason for their high price is the difficulty of the chemical separation of the individual elements of this group.

Represented under this common name are La, Ce, Pr, Nd, Il, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, and Lu, with atomic numbers of 57 to 71.

II. Glass Decolorization with Cerium and Neodymium

The first use of rare earths in ceramic materials was suggested by Drosbach,[1] who recommended that glass be decolorized by a mixture of rare earths. The cerium oxide in this mixture has an oxidizing effect on the divalent iron, whereas the neodymium compensates for the remaining yellowish color. This combination, in fact, for chemical and physical reasons, is the best decolorizer known at the present time. Price considerations and the difficulty of inaugurating the use of a new decolorizer, however, prevent its general application. Arsenic must not be used with this decolorizer because even traces of it would impair the color of the glass. According to Kohl,[2] a similar effect is obtained in ivory porcelain in which the replacement of manganese by cerium gives rise to a very pure color although the effect may not be caused by the oxidation of iron. With titanium oxide, cerium dioxide produces a beautiful yellow color in glass.

III. Ultraviolet Absorption of Cerium Glass

Schott investigated CeO_2 as a glassformer, but only the discovery of the remarkable absorption of this colorless compound in the ultraviolet range by the Sanoskop Company[3] in Germany in 1912 and by Crookes[4] in 1913 in England increased its importance. In designing protective eyeglasses, it was desirable to replace dark-colored, ultraviolet-absorbing glasses, such as ferric oxide glasses, by a glass which was almost as colorless as regular ophthalmic glass. These Crookes' glasses, which contained 2 to 4% of CeO_2, found general use in England although they were originally unsuccessful in Germany. They are known by this name in the United States and by the name of Dichroyal and Filtrays in France. Pure CeO_2 is not always necessary in these glasses and raw materials that contain La_2O_3, Nd_2O_3, Pr_2O_3, and Y_2O_3 are being used. The use of cerium oxide in combination with ThO_2, ZrO_2, and BeO for very viscous glasses of higher expansion than silica has been suggested by Pirani.[5]

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[1] P. Drosbach, "Decolorization of Glass with Rare Earths," Ger. Pat. 103,441 (1896).

[2] (*a*) Hans Kohl, "Red and Yellow Pigments in Ceramics: II, Yellow Colors, with Special Reference to Ivory Porcelain," *Ber. deut. keram. Ges.*, **17** [12] 597–622 (1936); *Ceram. Abs.*, **16** [8] 232 (1937).

(*b*) Hans Kohl, "Opacifiers and Their Economical Use," *Emailwaren-Ind.*, **15** [29–30] 199–202; [31] 207–10; [32] 215–19 (1938); *Ceram. Abs.*, **18** [4] 94 (1939).

[3] Sanoskop G.m.b.H., "Ultraviolet-Absorbing Glasses," Ger. Pat. 308,075, April 4, 1912.

[4] W. Crookes, "Preparation of Eye-Preserving Glass for Spectacles," *Trans. Roy. Soc.* [London], **A214**, 1 (1914).

[5] (*a*) M. Pirani, "Refractory Glass," U. S. Pat. 2,219,332, October 29, 1940; *Ceram. Abs.*, **20** [1] 16 (1941).

(*b*) M. Pirani and J. H. Partridge, "Vitreous Body," U. S. Pat. 2,219,331, October 29, 1940; *Ceram. Abs.*, **20** [1] 22 (1941).

IV. Glasses Colored with Neodymium and Praseodymium

An addition of some neodymium oxide improves the selenium pink,[6] and cerium and titanium oxides impart to the glass a beautiful yellow color and remarkable brilliance. One of the most beautiful glass colors, however, and the only bright purple known in glass is obtained by the use of pure neodymium oxide. Neodymium glasses were first prepared on a large scale by Weidert.[7(a)] Their specific absorption, consisting of several sharp bands[7(b)] in visible and in infrared wave length, has found several interesting applications.

In concentrations of 4 to 5%, neodymium is found in the famous purplish-blue glasses (Alexandrite glasses) of Bohemia, which were developed by L. Moser. The main absorption band of aqueous solutions of neodymium is in the yellow-green part of the spectrum. The same absorption band is present in this glass, and the visible spectrum is divided abruptly into a blue part and a red part. This peculiarity of the absorption spectrum causes different color sensations, depending on whether the light passes through thick or thin layers of glass. Because of this effect,[8] the Moser glasses of Bohemia exhibit a play of blue and red colors along the changing wall diameter in which they are purposely formed.

The discontinuity of the light transmission between red and blue enables the user of such a glass to distinguish certain colors more easily, especially red and green.[9] Such glass may be used either as an eyeglass or as a lamp shade. Neodymium glasses of higher concentration are also used for navigation and aviation,[10] and they have been recommended to illuminate recreation and sales rooms by filters or colored bulbs.[11] This glass has been called Neophane (Philiphan in Holland).

Long[12] has made use of the intensive absorption by neodymium glass in the yellow part of the spectrum in another invention. Windows made of neodymium glass will render a room illuminated by sodium lamps invisible to attacking aircraft. He has also applied sodium light in combination with a neodymium glass in a device for hospitals, by means of which an operation may take place in an illuminated room and the progress of the operation may be checked on a fluorescent X-ray screen. In place of the usual heavy lead glass for the absorption of the X rays, a lead glass colored with neodymium oxide is used in combination with the screen. When the room is lighted by sodium lamps, the screen looks dark under the neodymium glass and scintillates only when excited by X rays.

V. Optical Glasses

Praseodymium oxide gives a green color although this color may be obtained just as well by cheaper oxides. With neodymium oxide, it gives grayish tints.[13]

The use of lanthanum oxide in optical borate glasses of high refractive index and low dispersion in combination with ThO_2 and tantalum oxide has been suggested in the patents of Morey and Eberlin.[14] (See p. 138, this issue.)

VI. Enamels and Glazes

In Europe, cerium oxides are being used for the opacification of enamels.[15] Additions as small as 4% may furnish a reflectance of 75 which corresponds to the opacity of enamels that are clouded with tin oxide. According to Kohl,[2] 1.5% of CeO_2 corresponds to 6% of SnO_2, 7% of antimonate, or 4.5% of gas opacifier. Cerium oxide is also used in combination with gas opacification.[2]

The influence of the crystalline structure of cerium oxide and its solubility in various enamels have been discussed recently by Paquet.[15(b)] The opacifying phase is the face-centered cubic CeO_2. The high refractive index of 2.3 explains why only a small amount of CeO_2 is necessary to replace tin oxide, which has a refractive index of 2, in the mill. There is a considerable amount of solubility in the enamel frit, and it may also cause a yellow hue. Its use is therefore restricted to mill opacification, and frit opacification is excluded. Calcination with other oxides and salts is beneficial even if the cerium oxide content of the opacifier is reduced by the addition of considerable amounts of these compounds; for example, 35% of MgO may be used. The enamel industry in France has tried other oxides, but the results are unknown. The addition of 3 to 4% of

[6] J. Löffler, "Selenium Pink as a Glass Color and Its Modification by Rare Earths," *Glastech. Ber.*, **15** [10] 389–93 (1937); *Ceram. Abs.*, **17** [5] 172 (1938).

[7] (*a*) F. Weidert, "Absorption Spectrum of Didymium Glasses with Various Compositions of the Base Glass," *Z. wiss. Phot.*, **21**, 254–64 (1921–1922); abstracted in *Glastech. Ber.*, **6**, 596 (1928–1929).

(*b*) K. Rosenhauer and F. Weidert, "Spectral Absorption of Neodymium Glasses," *Glastech. Ber.*, **16** [2] 51–57 (1938); *Ceram. Abs.*, **17** [10] 326 (1938).

[8] V. Čtyroký, "Glasses Colored with Neodymium Oxide and Vanadium Oxide," *Sklářské Rozhledy*, **15** [5–6] 94–128 (1938); *Glashütte*, **69** [17] 305–306; [18] 320–25; [19] 337–40 (1939); *Glastech. Ber.*, **18** [1] 1–7 (1940); *Ceram. Abs.*, **19** [6] 136 (1940).

[9] W. Ströble, "Effect of Light Filters, Especially of Neodymium-Containing Glasses on Color Sensation," *Mitt. Opt. Inst. Tech. Hochschule*, Berlin, 1937; abstracted in *Physik. Ber.*, **19** [12] 1266 (1938); *Ceram. Abs.*, **17** [11] 350 (1938).

[10] F. Weidert and Hans Löffler, "Neodymium Glasses Used in Navigation and Aviation," U. S. Pat. 2,219,122, October 22, 1940; *Ceram. Abs.*, **20** [1] 16 (1941).

[11] (*a*) C. Schaefer and J. Rosemann, "Influence of Neophane Glass on Visibility of Interference Patterns in White Light," *Z. tech. Physik.*, **20** [7] 193–98 (1939); *Ceram. Abs.*, **18** [11] 294 (1939).

(*b*) F. J. Bouma, "Neophane Glass (Philiphan)," *Philips Tech. Rev.*, **3** [2] 27–29 (1938).

[12] B. Long, "Röntgen-Ray Apparatus," Brit. Pat. 450,513, July 15, 1936; *Chem. Abs.*, **31** [1] 41–42 (1937).

[13] V. Čtyroký, "Importance of Rare Earths for Glass Industry," *Sklářské Rozhledy*, **15**, 23–33 (1938); *Ceram. Abs.*, **17** [8] 274 (1938).

[14] (*a*) L. W. Eberlin, "Rare Earth Borate Compositions," U. S. Pat. 2,206,081, July 2, 1940; *Ceram. Abs.*, **19** [9] 213 (1940).

(*b*) G. W. Morey, "Optical Glass," U. S. Pat. 2,150,694, March 14, 1939; *Ceram. Abs.*, **18** [5] 127; [11] 297 (1939).

[15] (*a*) H. S. Cooper, "Enamel Composition," U. S. Pat. 1,510,829, October 7, 1924; *Ceram. Abs.*, **4** [1] 5 (1925).

(*b*) M. Paquet, "Applications of Cerium, Zirconium, and Titanium Compounds to the Production of Enamels on Metals and Ceramic Ware," *Compt. Rend. 18me Congr. Chim. Ind.*, **1938**, 1096–1107; *Ceram. Abs.*, **18** [11] 291 (1939).

Al_2O_3 or of ZrO_2 or of both these oxides to the frit makes the cerium oxide less soluble. Paquet states that the opacification by CeO_2 cannot be the result only of the simple phenomenon of the difference of refraction. He was unable to reproduce the opacification in an organic resin as a model of the same index as the frit.

Owing to the present high cost of cerium oxide as compared with that of other opacifiers, its application is still limited. Only in France has the extreme activity of the producers of rare earth materials succeeded in securing a place for the cerium opacifier in enamels.

Melzer[16] describes the use of 3% of CeO_2 in bleaching cobalt ground coats and its application in cobalt-free ground coats instead of antimony.

The replacement of the expensive pure cerium oxide by mixtures with rare earths in the opacification of glazes has been studied.[17] The comparison was made on the basis of equal cerium oxide content. Such mixtures impart brownish hues and increase the viscosity in a manner similar to that of alumina. Although opacification with these mixtures is inferior to that of the pure oxide, some colored glazes could be developed. The crystals of ZnO glazes, moreover, are retarded by the addition of the rare earth mixture.

The residues of thorium nitrate may be used in opaque lead glazes on terra cotta. These residues contain cerium, neodymium, praseodymium, lanthanum, and yttrium. When neodymium and praseodymium were separated for the application of neodymium, pure lanthanum oxide was obtained. The manufacturers of rare earths have encouraged research on the use of this oxide, but few possible applications are known. Arndt and Puetter[18] have suggested that ZnO be replaced partly by lanthanum oxide. When Schuen[19] tried the addition of various chlorides to improve the salt glaze on earthenware of high alumina content, lanthanum chloride was found to yield good results, but its high price still prevents its practical application.

Giusca[20] has tried to replace the alumina of cordierite by rare earths, but he invariably obtained the simple silicates because of the higher coordination numbers of neodymium, lanthanum, and yttrium as compared to that of aluminum.

The use of cerium oxide as a refractory material was investigated by von Wartenberg and Eckhardt[21] who found that it could be used for extreme conditions. Pure CeO_2 cannot be reduced, and mixtures that contain ThO_2 melt above 2600°C. These authors have also included the systems La_2O_3–ZrO_2 and La_2O_3–MgO in their work.

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[16] W. Obst cites Melzer in "Recent Experience with Cerium Oxide in the Enamel Industry," *Emailwaren-Ind.*, **14** [44–45] 303–304 (1937).

[17] B. W. Merwin, "Cerium Oxide and Rare Earth Oxide Mixture in Glaze," *Jour. Amer. Ceram. Soc.*, **20** [3] 96–98 (1937).

[18] E. Arndt and K. E. Puetter, "Importance of Lanthanum," *Angew. Chem.*, **51** [27] 463–64 (1938).

[19] W. Schuen, "Vitrified Brick and Salt Glaze," *Keram. Rundschau*, **47** [14] 147–49 (1939); *Tonind. Ztg.*, **63** [31] 357–59 (1939); *Ceram. Abs.*, **19** [4] 92 (1940).

[20] D. Giusca, "Crystal Chemistry of Simple Rare Earth Silicates," *Bul. Laboratorului Mineralogie Universitatii Bucuresti*, No. 1, 13 pp. (1934).

[21] H. von Wartenberg and K. Eckhardt, "Fusion-Point Diagrams of Highly Refractory Oxides: VIII, Systems with CeO_2," *Z. anorg. allgem. Chem.*, **232** [2] 179–87 (1937); *Ceram. Abs.*, **19** [4] 100 (1940).

VARIABLE FACTORS IN ACCELERATED AUTOCLAVE CHEMICAL DURABILITY TESTS*

BY J. S. OWENS AND E. C. EMANUEL

ABSTRACT

An investigation was conducted of certain factors which influence the reproducibility of autoclave chemical durability tests. These factors include the method of preparation and the purity of the water used, the rinsing procedure, the volume of solution used, and variations in the test temperature and in the duration of the different periods of the heat cycle. The effect of the internal area of the container on the test results was determined, and suggestions are presented for increasing the reliability of the tests.

I. Introduction

A considerable amount of effort in various laboratories has been devoted recently to the development of accelerated, alkali extraction tests of the chemical durability of glass containers and to the possible correlation of the results of such tests with actual practice. Reproducible results have been difficult to obtain with any one test, particularly if the data obtained in different laboratories are compared.

The various accelerated tests which have been proposed and investigated include the action on a crushed glass sample or on the internal surface of a container by dilute acid, water, or dilute alkali at 80° to 138°C. for periods from 1 to 24 hours.

A chemical durability test, which comprises the determination of the alkali extracted from the internal

* Presented at the Forty-Third Annual Meeting, The American Ceramic Society, Baltimore, Md., April 1, 1941 (Glass Division). Received June 7, 1941.

surface of the container by water at 120°C. for 1 hour, has been proposed and is in actual commercial use. In view of the commercial importance of this test and of the difficulty experienced in obtaining reproducible results, either in one or among the different laboratories, an investigation of the effects of certain variable factors in the test has been made in an effort to improve the precision and accuracy.

The pertinent factors chosen for investigation were (1) the preparation and condition of the distilled water, (2) the degree of rinsing before filling the bottles, (3) the internal area of the bottle, (4) the volume of water introduced into the bottles, and (5) the variable temperature and time of heating, test, and cooling periods.

To establish uniform conditions, these tests were made with 8-oz. flint bottles which were manufactured approximately two weeks before the beginning of the tests. The internal area of the samples averaged 291 sq. cm.

II. Chemical Durability Test Procedure

The normal procedure employed in this laboratory for the test (hereafter referred to as the "normal test") was as follows:

Doubly distilled water was used, which may be prepared by redistilling the usual laboratory-distilled water in an all Pyrex-brand still fitted with No-Lub ground-glass joints. The proper portion of the distillate was collected and boiled in a Pyrex-brand flask to approximately three fourths of its original volume to remove any residual gases. The purity of the boiled water was tested by titrating a 100-ml. portion while hot (approximately 70° to 80° C.) with 0.02 *N* H_2SO_4, using 2 drops of methyl red indicator (0.2 gm. of methyl red in 100 ml. of 95% ethyl alcohol). The amount of acid required was about −0.02 to +0.02 ml. The water was stored in a sealed 5-gallon Pyrex-brand bottle, fitted with a soda-lime tube and a Pyrex-brand siphon, from which it was withdrawn for use without reboiling.

Each 8-oz. bottle to be tested was rinsed with 40 ml. of the prepared water. Immediately after introducing the water, the bottle was covered with tinfoil, shaken three times so that the entire internal surface was washed, and emptied. Each bottle was then filled to its rated capacity with the prepared water at room temperature and loosely capped with tinfoil crimped over the neck and retained in place by rubber bands. Two 250-ml. Pyrex-brand Erlenmeyer flasks, which were similarly rinsed, filled with 237 ml. of water, and capped, were placed in the autoclave with the bottles to serve as blank controls.

The closed, gas-heated autoclave,* containing distilled water to a depth of two inches, was heated to 100°C. with the vent valve open. The bottles were introduced, and the closed autoclave, with the vent valve open, was heated to 100°C. in 15 minutes. The valve was closed, and the autoclave temperature was raised uniformly to 120°C. in exactly 15 minutes. The temperature was maintained at 120°C. for 60 minutes by regulating the flame beneath the autoclave and was then lowered to 100°C. in exactly 30 minutes by regulating the vent valve.

The bottles were removed from the autoclave and were immediately shaken three times. One hundred milliliters of the water from each bottle and from each of the blanks were titrated with 0.02 *N* H_2SO_4 to a methyl red end point while hot in a 250-ml. Pyrex-brand Erlenmeyer flask. The net titer, after correction for the blank, may be expressed as milligrams of NaOH per liter.

III. Experimental Results

(1) Preparation of Distilled Water

Experiments were made to compare the properties of the water, which was obtained in a Pyrex-brand still by a single distillation of tap water and by redistillation of laboratory-distilled water, and to test the efficacy of certain reagents added to the still boiler. The reagents used were alkaline permanganate solution, which contained 200 gm. of potassium hydroxide and 8 gm. of potassium permanganate per liter, and phosphoric acid, which was prepared by heating c.p. 85% phosphoric acid to white fumes.[1] One hundred milliliters of the alkaline permanganate solution were added to 4 liters of water in the still boiler, and additions of 0.1 ml. and of 6.4 ml. of phosphoric acid to 4 liters of water in the boiler were employed.

* A satisfactory autoclave heated by other means may be used.

[1] (*a*) C. B. Clevenger, "Rapid and Convenient Method for Preparation of Conductivity Water," *Ind. Eng. Chem.*, **11**, 964–66 (1919).
(*b*) S. B. Ellis and S. J. Keil, "Purification of Water and Its pH Value," *Jour. Amer. Chem. Soc.*, **57**, 2145–49 (1935).

TABLE I

PREPARATION OF DISTILLED WATER

Water in boiler	Distillate fraction (%)	Reagent in boiler	NH_3 (p.p.m.)		PO_4 (p.p.m.)		Titer (mg. of NaOH/liter)		Chemical durability test (mg. of NaOH/liter)
			Before boiling	After boiling	Before boiling	After boiling	Before boiling	After boiling	
Distilled	0–25	None	3.0	2.0	Nil†	Nil†	0.0	0.96	10.00
	25–50		0.6	Nil*	"	"	.32	.40	10.48
	50–75		Nil*	"	"	"	− .32	.16	11.44
	75–90		0.01	"	"	"	− .48	.16	10.64
Distilled	0–25	H_3PO_4‡	Nil*	"	0.3	"	− .16	.0	10.24
	25–50		"	"	0.16	0.06	− .08	.0	10.72
	50–75		"	"	Nil†	Nil†	− .08	.0	10.08
	75–90		"	"	"	"	− .16	.0	9.60

* Nil < 0.01 p.p.m. † Nil < 0.02 p.p.m. ‡ The amount of H_3PO_4 is 0.1 ml. to 4 liters of water.

The initial 90% of the distillate in each method of preparation was collected in four fractions. The properties of the fractions were compared (1) by analysis for ammonia and for phosphate, respectively, (2) by carbon dioxide content (determined relatively by difference in titer of a 100-ml. aliquot before and after boiling), (3) by degree of neutrality of the boiled distillate, and (4) in some cases by results of autoclave chemical durability tests on the usual 8-oz. bottles.

The best water from the standpoint of the uniformly low ammonia and carbon dioxide content, the practical neutrality of the distillate, and the uniformity of the chemical durability values obtained with the different fractions is produced by redistilling laboratory-distilled water to 4 liters of which 0.1 ml. of phosphoric acid has been added. Doubly distilled water of satisfactory purity may be prepared without the addition of phosphoric acid provided a larger portion of the distillate

TABLE II

EFFECT OF METAL SALTS

Compound	Concentration of metal (p.p.m.)	Net titer (mg. of NaOH/liter)	Deviation from normal test (mg. of NaOH/liter)	Minimum significant concentration of metal (p.p.m.)	Net titer of solution* (mg. of NaOH/liter)
None†		10.32	0		0.00
$CuSO_4$	0.00009	10.56	+0.24	0.002	.00
	.0009	10.00	−0.32		.00
	.009	9.44	−0.88		.00
	.9	3.80	−6.52		.00
$FeSO_4$	.00075	9.91	−0.41	.002	−0.04
	.0075	9.75	−0.57		−0.04
	.075	9.68	−0.64		−0.20
	.75	8.00	−2.32		−1.08
$Fe_2(SO_4)_3$	.00058	10.24	−0.08	.01	+0.12
	.0058	10.40	+0.08		+0.04
	.010	9.84	−0.48		+0.04
	.075	9.84	−0.48		−0.12
	.10	10.00	−0.32		+0.04
	.58	7.52	−2.80		
	.75	6.88	−3.44		−1.40
	58.0	1.60	−8.72		
$MgCl_2$	0.01	10.16	−0.16	.04	−0.04
	0.04	9.84	−0.48		+ .04
	0.1	8.80	−1.52		+ .04
	0.4	9.20	−1.12		+ .04
	1.0	8.24	−2.08		+ .04
	4.0	7.60	−2.72		+ .12
NH_4NO_3	0.03	10.00	−0.32	.04	.00
	0.3	8.56	−1.76		− .08
	3.0	2.88	−7.44		− .40
NH_4Cl	0.003	10.24	−0.08	.06	.00
	0.03	10.26	−0.06		− .32
	0.1	9.20	−1.12		.00
	0.3	8.72	−1.60		.00
	3.0	3.52	−6.80		− .16
	30.0	3.12	−7.20		− .40
$(NH_4)_2SO_4$	0.03	10.24	−0.08	.12	− .08
	0.3	9.60	−0.72		− .08
	3.0	2.96	−7.36		.00
$CaCl_2 \cdot 2H_2O$	0.04	10.50	+0.18	.20	− .04
	.1	10.72	+0.40		+ .04
	.2	10.48	+0.16		− .12
	0.4	11.50	+1.18		− .04
	4.0	15.34	+5.02		− .04
	40.0	19.92	+9.60		− .20
$Na_2SiO_3 \cdot 9H_2O$	0.02	10.40	+0.08	0.21	+0.16
	0.2	10.80	+0.48		+0.48
	2.0	12.49	+2.17		+3.20
	20.0	28.96	+18.64		+34.72
NaCl	0.023	10.08	−0.24	2.3	0.00
	0.23	10.08	−0.24		.00
	2.3	9.84	−0.48		.00
	23.0	8.96	−1.36		.00

* Titer of the solution minus titer of water used to prepare solution.
† Normal test.

is discarded, which decreases the efficiency of the still.

Table I shows a representative comparison of the doubly distilled water prepared with and without the addition of phosphoric acid to the still boiler.

All of the water used subsequently was prepared by the addition of phosphoric acid to the still boiler. The titer of this water, which was determined immediately after being boiled in 3-liter Pyrex-brand flasks, did not exceed the limits of −0.16 to +0.16 mg. of NaOH per liter. It was found that aliquots of the same water, boiled in different Pyrex-brand Erlenmeyer flasks of 1-liter capacity, might yield significantly different titers. To avoid the effect of this condition, all distillates were collected in 6-liter Pyrex-brand flasks, transferred to 3-liter Pyrex-brand flasks in which the water was boiled, and the boiled water was then introduced while hot into a 5-gallon, sealed Pyrex-brand bottle, which was equipped with a soda-lime tube. The bottle was also fitted with a Pyrex-brand siphon containing a short rubber connection through which the water was introduced into and withdrawn from the bottle without contamination by carbon dioxide. The different lots of water used in all of the tests thus had essentially identical properties. The titers of the Pyrex-brand blanks in the autoclave tests varied from zero to 0.24 mg. of NaOH per liter.

(2) Effect of Carbon Dioxide in Water

In order to determine the effect of carbon dioxide that is present in the water in the bottles before their introduction in the autoclave or in the solutions at the time of titration, the following experiments were made:

(1) Bottles, filled as usual and capped loosely with tinfoil, were allowed to stand at room temperature in the laboratory atmosphere for periods up to 180 minutes. The autoclave test results obtained with these bottles were compared with those of bottles which contained unboiled as well as CO_2-saturated water.

(2) After a normal autoclave treatment of duplicate bottles, the different bottles, covered with tinfoil, were allowed to stand at room temperature following their removal from the autoclave and were titrated at successive intervals up to 160 minutes to determine the effect of temperature and of absorption of carbon dioxide on the titer obtained. After the first titration, the solutions were boiled and retitrated.

(3) To determine whether carbon dioxide is effective in reducing the alkali extraction by the water or in affecting the titration itself, a chemical durability test was made with duplicate bottles and with Pyrex-brand Erlenmeyer flasks containing solutions of normal prepared water, CO_2-saturated water, 0.0003 *N* Na_2CO_3, and 0.0006 *N* NaOH. After the autoclave treatment, an aliquot of the solution from each container was titrated, boiled, and retitrated. Another aliquot was boiled and then titrated.

The data obtained in these experiments show that (1) no appreciable amount of carbon dioxide is absorbed by the filled bottles before or after autoclaving for periods up to 3 hours; (2) carbon dioxide in the water before the test decreases both the amount of alkali extracted in the autoclave and the titration value; (3) a small amount of carbon dioxide apparently is present in the containers after the autoclave treatment, regardless of whether they originally contain water, dilute Na_2CO_3 solution, or dilute NaOH solution; and (4) the temperature of the solution, when titrated, has no significant effect on the titer obtained.

(3) Effect of Metal Salts in Water

Experiments were made to determine the effect on the result of the otherwise normal test of different concentrations of various metal salts in the water used in the bottles and in the Pyrex-brand blanks. To 227 ml. of water in each bottle and Pyrex-brand blank were added 10 ml. of a solution which contained the proper concentration of the salt to give the desired final concentration in the container.

Table II shows (1) the compounds tested in the order of decreasing effect on the chemical durability test, (2) the quantitative effect of each compound on the test, (3) the minimum concentration of each metal which produced a significant effect (±0.5 mg. of NaOH per liter, just greater than the normal experimental variation) on the test, and (4) the net titer of a duplicate of each solution used in the bottles.

These data show that small concentrations of metals in the water may cause considerable error in the test results. Not only do the minimum concentrations of the various metals which will produce a significant effect on the test differ in order of magnitude, but the negative radical in the compound may also exercise appreciable influence.

Table II shows that, of the compounds used, only $CaCl_2 \cdot 2H_2O$ and $Na_2SiO_3 \cdot 9H_2O$ increased the test results.

The titrations of duplicates of the solutions used gave no indication, except for impurity concentrations much greater than the minimum significant values, of the effects produced on the autoclave test.

The replacement of the tinfoil covers of the bottles with inverted Pyrex-brand beakers was found to increase the autoclave test results by 0.30 mg. of NaOH

TABLE III

EFFECT OF RINSING PROCEDURES

No. of rinses	Method of rinse	Net titer (mg. of NaOH/liter)	Deviation from normal test (mg. of NaOH/liter)
1	H_2O 40 ml.; 3 shakes*	10.32	0.00
None	None	11.20	+ .88
None	Bottles blown out with filtered compressed air	10.96	+ .64
2	H_2O 40 ml.; 3 shakes	9.76	− .56
4	H_2O 40 ml.; 3 shakes	9.92	− .40
1	H_2O 40 ml.; 5 minutes of thorough rinse	9.92	− .40
1	Full bottle; standing 10 minutes	9.60	− .72
1	Full bottle; shaken 10 minutes	9.28	−1.04

* Normal test.

per liter. This deviation is within the normal experimental error of approximately ±0.40 mg. of NaOH per liter.

(4) Effect of Rinsing

The effects of different rinsing procedures used before the bottles were filled for the otherwise normal test are shown in Table III. These bottles were approximately 9 weeks old and were stored uncapped before use in paper cartons.

These data show the importance of a uniform rinsing procedure before filling the bottles and of performing the rinsing quickly to prevent the water from standing in the bottles for any length of time.

With increasing age and weathering of the bottles, this effect of rinsing would be expected to become increasingly important.

(5) Effect of Internal Area and Degree of Fill of Bottles

(*A*) *Measurement of Internal Area:* The following rapid method for measuring the internal area of a bottle was developed by the laboratory staff. The bottle was partially filled with a 20% (by volume) solution of methyl methacrylate in toluol and was slowly swirled until the entire internal area was coated with the solution. The excess solution was drained out and low-pressure compressed air was blown into the bottle until the coating just ceased to be tacky. A second coating was given in similar fashion and was thoroughly dried by compressed air. The bottle was then externally scratched in several places, cracked by a severe thermal shock, and removed from the resin coating. The coating was cut so that it would lie flat, traced, and the tracing was planimetered. The results on duplicate bottles showed that the error in the measured area, in general, should not exceed ±3%.

(*B*) *Effect of Internal Area:* In order to determine the influence of the internal area of the container on the results of the autoclave test, tests were made on a series of bottles manufactured simultaneously in two furnaces from the same batch formula. The ten types of bottles varied in nominal capacity from 0.5 to 32 ounces and represented square, rectangular, cylindrical, and flask shapes. As the internal area and nominal capacity of the bottle increased, the total amount of alkali extracted was found to increase and the concentration of alkali in the liquid extract decreased (see Fig. 1, curves (1) and (2), respectively). This conclusion agrees with Turner's results[3] on the 5-hr. boiling-water test. The alkali extracted per unit internal area decreased about 30% for a 13-fold increase in area. Inasmuch as the conditions of manufacture and of annealing were not identical for the bottles of different size, the cause of this decrease in the alkali extracted per unit area could not be determined.

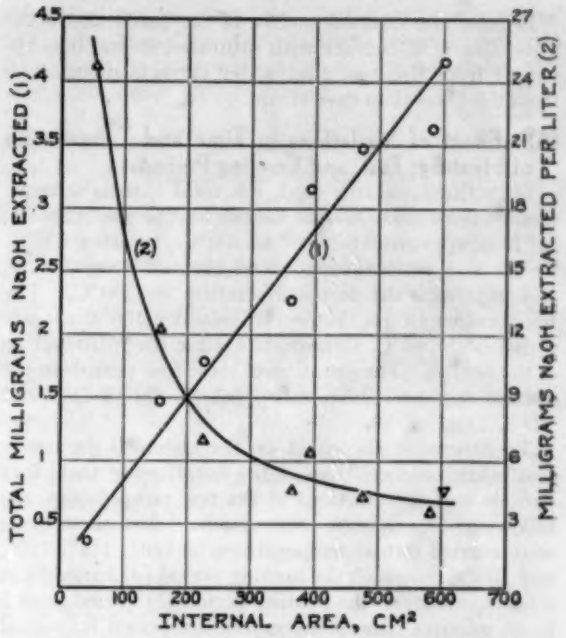

FIG. 1.—Effect of internal area of container on alkali extracted.

These results show the important effect of the internal area of the container on the results of the chemical durability test and that this area must be taken into account in the evaluation of the durability of the glass of any particular type of bottle. For essentially the same formulation, the amount of alkali extracted per unit of internal surface seems to be roughly constant for containers of different sizes and shapes.

(*C*) *Effect of Volume of Water in Container:* Table IV shows the effect of the amount of water placed in the bottle for the chemical durability test.

These data show that, with increasing volume of water in the bottle, (1) the total amount of alkali extracted and the amount of alkali extracted per unit of total internal area increase, and (2) the concentration of the liquid extract and the amount of alkali extracted per unit of internal area covered by water decrease.

[3] W. E. S. Turner, *et al.*, "Basis of Standard Test for Chemical Durability of Glass Bottles," *Jour. Soc. Glass Tech.*, **19**, 171–88 (1935); *Ceram. Abs.*, **16** [7] 200 (1937).

TABLE IV

EFFECT OF VOLUME OF WATER IN BOTTLE

Total internal area (cm.²)	Internal area covered by water (cm.²)	Vol. of water in bottle (ml.)	Net titer (mg. of NaOH/liter)	Alkali extracted (mg. of NaOH)		
				Total	Per cm.² of surface covered by water	Per cm.² of total surface
291	142	120	14.20	1.70	0.0120	0.0059
	179	160	12.00	1.92	.0107	.0066
	219	198	10.92	2.16	.0099	.0074
	265	237	10.40	2.47	.0093	.0085

Because the internal surface of the bottle above the water line is in contact with saturated steam, it is apparent from this test that water extracts more alkali from the glass than does steam.

(6) Effect of Variations in Time and Temperature of Heating, Test, and Cooling Periods

With the apparatus used, the total autoclave treatment may be considered to include four periods, namely, (*a*) from approximately 80° to 100°C., (*b*) from 100°C. to the test temperature, (*c*) at the test temperature, and (*d*) from the test temperature to 100°C. The temperature of the autoclave, initially 100°C., is approximately 80°C. immediately after the introduction of the bottles. The duration of these four periods in the normal test are, in minutes, (*a*) 15, (*b*) 15, (*c*) 60 at 120°C., and (*d*) 30.

To determine the effect on the chemical durability test results of variations in the durations of these four periods and of variations in the test temperature, the following experiments were made. Autoclave tests were carried out at temperatures of 110°, 115°, 120°, and 130°C. in which the heating period (*a*) varied from 8 to 15 minutes; the heating period (*b*) varied from 5 to 45 minutes; the test-temperature period (*c*) varied from 0 to 180 minutes; and the cooling period (*d*) varied from 5 to 45 minutes. These extreme variations were not used for the tests at each temperature. Tests were also made at 100°C. with a heating period (*a*) of 15 minutes and with period (*c*) varying from 180 to 480 minutes.

With the particular apparatus used, the temperature of the water in the bottles during the heating period was found to lag behind that of the autoclave steam by approximately 8 minutes. The manner in which the data were obtained, however, prevented this lag from appreciably affecting the calculated results.

It was also realized that the amount of alkali extracted per minute during each period was not constant but probably a function of the time. From the experiments described, however, the average apparent amount of alkali extracted per minute during each period (as determined by the autoclave temperature) of the test at each temperature was calculated so that the approximate effect on the test results of variations in the duration or temperature of these periods might be determined (see Table V).

These data were obtained from the solutions of simultaneous equations which were derived from the results of the different tests. Absolute accuracy is not claimed for these calculated alkali extraction values, but they are sufficiently precise to enable the effects of variations in the test procedure to be predicted and to show certain relations between tests made at different temperatures.

Some alkali undoubtedly is extracted during the heating from 80° to 100°C., but the amount apparently is negligible in comparison with that extracted at temperatures above 100°C.

Figure 2, plotted from the data in the fourth column of Table V, shows that when the effects of the heating and cooling periods are eliminated, the alkali extracted per minute varies logarithmically with the temperature from 100° to 130°C.

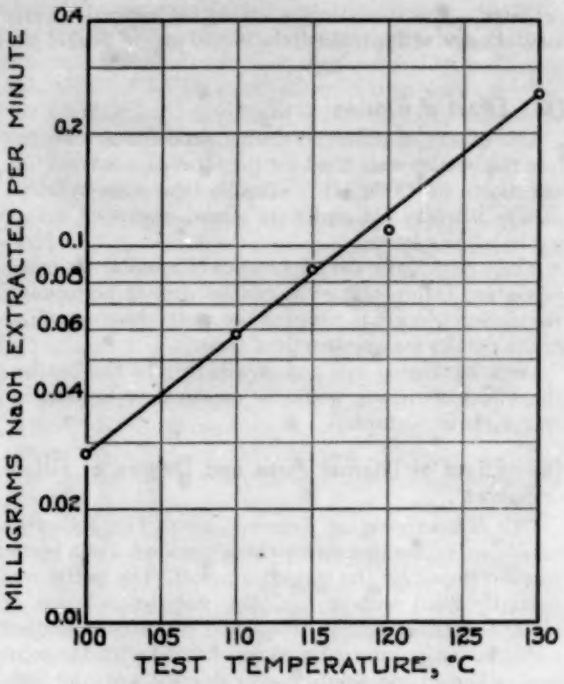

FIG. 2.—Relation between test temperature and alkali extraction per minute.

TABLE V

ALKALI EXTRACTED PER MINUTE DURING PERIODS OF TEST

Test temp. (°C.)	Alkali extracted per minute (mg. of NaOH/liter) Test periods			
	(*a*) 80°–100°C.	(*b*) 100°C. to test temp.	(*c*) At test temp.	(*d*) Test temp. to 100°C.
100	0		0.028	
110	0	0.056	.058	0.045
115	0	.046	.086	.058
120	0	.072	.110	.084
130	0	.078	.252	.120

Figure 3 is a plot of the calculated time required at each test temperature to extract 10.4 mg. of NaOH per liter from the internal surface of the 8-ounce bottle used (1) under the usual test conditions, which include heating and cooling periods, and (2) at the constant test temperature only. The logarithmic relationship between the temperature and the time required for a constant amount of attack confirms the conclusion of Lyle and Sharp[3]. The increasing importance, moreover, of the alkali extraction during the heating and cooling periods with increasing test temperature is clearly shown. As the total amount of attack increases, curve (1) tends to become more nearly parallel to curve (2).

[3] A. K. Lyle and D. E. Sharp, "Some Observations on Chemical Durability of Glass." Presented at the Autumn Meeting of the Glass Division, Hershey, Pa., September 6, 1940.

These data reveal that a variation of 1 minute, at the normal test temperature of 120°C., from the usual period (ϵ) of 60 minutes will produce approximately 1% error in the test result, whereas a deviation of 1° from 120°C. over the entire period of 60 minutes will cause an error of about 5%. Inasmuch as the temperature is the more difficult factor to control, this result indicates the amount of care that must be exercised to obtain precise results. Because, in the experience of the writers, no great reliability may be placed in the accuracy of an autoclave pressure gauge over extended periods of time, this result also shows the necessity for frequent recalibration of the gauge, or better, the use of an accurate autoclave thermometer for control purposes.

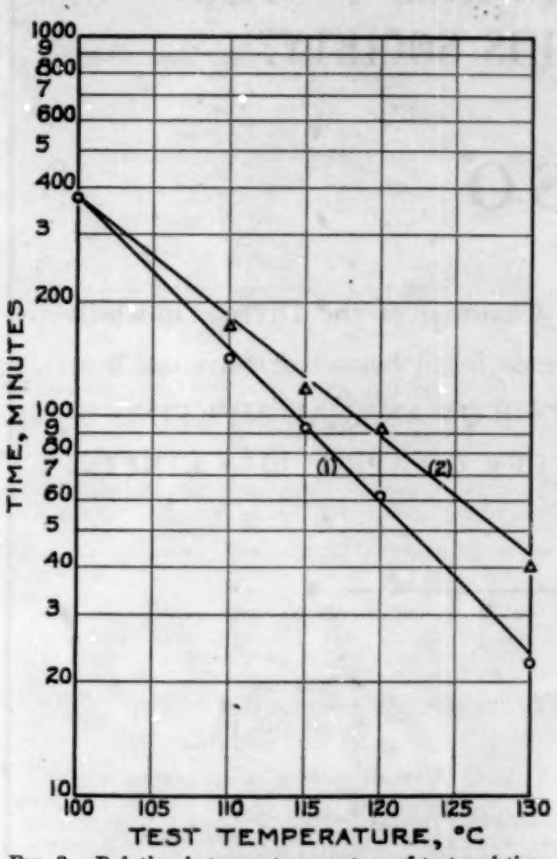

FIG. 3.—Relation between temperature of test and time required to extract constant amount of alkali.

(7) Precision of Test

The precision of the normal test was determined at the conclusion of this investigation by five tests made over a period of 11 days on 8-oz. bottles taken from the same carton. One lot of water was prepared for tests Nos. 1 and 2, a second lot was prepared for tests Nos. 3 and 4, and a third lot was prepared for test No. 5 (see Table VI).

TABLE VI

PRECISION OF NORMAL TEST

Bottle No.	Test No. 1	2	3	4	5	Avg.
	Alkali extracted (mg. of NaOH/liter)					
1	12.16	12.00	12.56	12.56	12.00	
2	12.16	12.16	12.24	12.48	12.00	
3	12.16	12.08	12.24	12.48	12.00	
4	12.32	12.24	12.24	12.56	12.40	
5			12.32	12.48		
Avg.	12.20	12.14	12.32	12.51	12.10	12.25

The average deviation from the mean in these tests amounted to 1.3%, and the maximum deviation from the mean was 2.5%.

IV. Summary

An investigation of certain variable factors in a commercial autoclave chemical durability test has shown that this test will yield results of good precision provided the details of the technique are carefully standardized.

The purity of the distilled water used is an important factor. Minute traces of certain elements and small amounts of carbon dioxide are detrimental. Doubly distilled water, prepared in an all Pyrex-brand still to the boiler of which a small amount of phosphoric acid is added, is satisfactory for use in the test.

A standardized rinsing method must be used. The internal area of the container must be taken into account in the evaluation of the chemical durability of the glass of any particular type of bottle. With an increasing volume of water placed in the bottle, the concentration of alkali in the liquid extract decreases, but the total amount of alkali extracted and that amount extracted per unit of total internal area increase.

The importance of careful control of the test temperature and of the times of the different periods of the heat cycle is pointed out.

By standardizing the technique, the normal error in the results of the tests on the 8-oz. bottles used may be reduced to no more than ±3% of the alkali extracted.

Acknowledgment

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CENTRAL TECHNICAL LABORATORY
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21—3

Abrasives

Abrasive paper and its manufacture. P. A. BENORD. *Papier-Ztg.*, **65**, 909–11, 932–33, 954–55 (1940); abstracted in *Chem. Zentr.*, **1941, I**, 257.—The manufacture of emery paper, etc., and the necessary mechanical equipment are described. See *Ceram. Abs.*, **20** [10] 229 (1941). M.V.C.

Electromagnetic feeler-gauge control. ANON. *Machinery* [N. Y.], **47** [1] 116 (1941).—The electromagnetic gauge for inspecting the measurements of small parts and for the automatic control of a grinding machine is described. Its application to the automatic control of diesinking operations is stressed. R.H.B.

Grinding steel-cutting carbide tools. GAYLORD G. THOMPSON. *Iron Age*, **148** [11] 41–44 (1941).—The grinding of carbide tools for cutting steel is a much more intricate procedure than the grinding of tools for cutting cast iron. Diamond wheels for rough grinding are considerably cheaper per tool service than silicon carbide wheels. Faulty practices in grinding are discussed and illustrated. E.H.McC.

Special grinder for finishing motor feet. ANON. *Iron Age*, **148** [2] 63 (1941).—The finishing operation can be handled by a shaper or a milling machine, but a surface grinder is the most satisfactory. Very little stock must be removed, and the feet need not be braced to avoid distortion. The surface grinder described is installed in works of the Reliance Electric & Engineering Co., Cleveland. The grinding head travels while the worktable remains stationary. E.H.McC.

SEPARATE PUBLICATION

Nonmetallic Minerals Needed for National Defense: II, Abrasives. LEO J. O'NEILL. *U. S. Bur. Mines Information Circ.*, No. 7168, 17 pp. Free.—The sources and supplies of artificial and natural abrasives, coated abrasives, and grinding wheels are covered with relation to their use in commodities needed for national defense. R.A.H.

PATENTS

Abrading machine. C. J. ASTROWSKI (General Electric Co.). U. S. 2,267,391, Dec. 23, 1941 (Aug. 2, 1940). C. O. JOHNSON. U. S. 2,269,311, Jan. 6, 1942 (Nov. 3, 1939).

Abrasive. J. H. BERGER (American Cyanamid Co.) Can. 401,264, Dec. 9, 1941 (May 7, 1940; in U. S. Sept. 13, 1939). WALTER W. DURANT (American Cyanamid Co.). Can. 401,462, Dec. 16, 1941 (May 14, 1940; in U. S. Jan. 9, 1940). G.M.H.

Abrasive article. EDWARD VAN DER PYL (Norton Co.). U. S. 2,270,209, Jan. 13, 1942 (May 20, 1939).

Abrasive article and method of making. G. P. NETHERLY, G. R. ANDERSON, AND B. S. CROSS (Minnesota Mining & Mfg. Co.). U. S. 2,269,415, Jan. 6, 1942 (Dec. 11, 1937).

Abrasive materials and method of manufacture. BABCOCK & WILCOX CO. Brit. 541,039, Nov. 26, 1941 (Feb. 11, 1939).

Abrasive tool. P. L. KUZMICK (J. K. Smit & Sons, Inc.). U. S. 2,268,663, Jan. 6, 1942 (Sept. 19, 1939).—An abrasive tool comprising a rigid support provided with a peripheral metal mesh ring.

Buffing machine. M. H. STEVENS. U. S. 2,269,133, Jan. 6, 1942 (April 9, 1940).

Coated abrasive article. G. P. NETHERLY, B. S. CROSS, AND G. R. ANDERSON (Minnesota Mining & Mfg. Co.). U. S. 2,269,416, Jan. 6, 1942 (May 20, 1941).

Combined shield and illuminating means for grinding wheels. S. A. BROWN (Brown-Brockmeyer Co., Inc.). U. S. 2,267,189, Dec. 23, 1941 (Jan. 11, 1940).

Disk abrading machine. A. N. EMMONS (Porter-Cable Machine Co., Inc.). U. S. 2,268,863, Jan. 6, 1942 (April 28, 1939).

Forming abrasive surfaces on high-speed grinding wheels. F. F. GILMORE AND F. W. KOTLER (F. F. Gilmore). U. S. 2,268,983, Jan. 6, 1942 (Aug. 2, 1940).

Grinding lathe and shaping tool bits. R. LINGHAM. Brit. 541,433, Dec. 10, 1941 (July 27, 1940).

Grinding machine. HARRY ARTER (Arter Grinding Machine Co.). U. S. 2,269,805, Jan. 13, 1942 (Feb. 29, 1940). ERICH BUCHHOLZ (Bauer & Schaurte). U. S. 2,268,694, Jan. 6, 1942 (July 12, 1939). C. G. FLYGARE AND J. I. GARSIDE (Norton Co.). U. S. 2,270,206, Jan. 13, 1942 (Nov. 4, 1939).

Grinding machine-table reciprocating mechanism. J. I. GARSIDE (Norton Co.). U. S. 2,268,389, Dec. 30, 1941 (May 14, 1940).

Grinding and polishing apparatus. C. M. GRIFFIN (Minnesota Mining & Mfg. Co.). U. S. 2,267,521, Dec. 23, 1941 (March 13, 1940). A. E. HAMILTON. U. S. 2,269,197, Jan. 6, 1942 (Sept. 20, 1939).

Grinding saws. J. A. ST. CLAIR (E. C. Atkins and Co.). U. S. 2,269,556, Jan. 13, 1942 (May 19, 1938).

Honing machine. W. M. FAIRBAIRN (Barnes Drill Co.). U. S. 2,268,028, Dec. 30, 1941 (Aug. 9, 1939).

Lapping machine. H. S. INDGE (Norton Co.). Can. 401,518, Dec. 16, 1941 (Feb. 8, 1940; in U. S. Feb. 8, 1939). G.M.H.

Lapping machines. L. HARTRIDGE. Brit. 541,703, Dec. 17, 1941 (Aug. 12, 1940).

Lapping valve plugs. G. F. SCHERER (Merco Nordstrom Valve Co.). U. S. 2,268,723, Jan. 6, 1942 (Nov. 12, 1938).

Machines for grinding internal screw threads. H. F. ATKINS. Brit. 541,263, Dec. 3, 1941 (Dec. 20, 1940).

Micro-lapping machine. H. S. INDGE (Norton Co.). Can. 401,517, Dec. 16, 1941 (Jan. 27, 1940; in U. S. Jan. 31, 1939). G.M.H.

Surface-grinding machine. H. A. SILVEN (Norton Co.). U. S. 2,269,697, Jan. 13, 1942 (Oct. 18, 1940).

Tool rests or work supports for emery-wheel grinders, etc. T. S. HARRISON & SONS, LTD., AND G. B. ASHTON. Brit. 540,850, Nov. 12, 1940.

Valve buffing and polishing machine. E. D. GEORGE AND H. M. BROWN (Wingfoot Corp.). U. S. 2,269,658, Jan. 13, 1942 (March 4, 1938).

Art and Archeology

China trade. JOSEPH DOWNS. *Bull. Metropolitan Mus. Art*, **36** [7] 156–58 (1941).—Several loans have enhanced the recent exhibition, "The China Trade and Its Influences," including a porcelain cake plate brought from China in 1796 as a present for Martha Washington. It was part of a tea set ordered by Andreas Everardus van Braam Houckgeest during his last visit to the court of Peking as a member of the embassy of the Dutch East India Co. He brought a large Oriental collection when he settled near Philadelphia to become an American citizen in 1784. A porcelain jar with basket-weave pattern, part of a five-piece garniture, was loaned by Mr. and Mrs. John DeWitt Peltz. It bears the monogram J M S, and was bought for John and Magdalena Douw Stevenson, according to a document dated April 23, 1798, which is also exhibited. A nest of porcelain barrels decorated with polychrome enamel was loaned by Mr. and Mrs. Arthur Talbot. See *Ceram. Abs.*, **21** [1] 3 (1942). A.A.A.

Connections between Egypt and Russia. M. A. MURRAY. *Antiquity*, **15** [60] 384–86 (1941).—A pottery tray of Tripolye culture from a site on the Dnieper River and a seated female pottery figure found in Russia closely resemble pottery soul houses and figures of the 13th dynasty of Egypt. Illustrated. A.B.S.

Craftsmen further south of the Rio Grande. MARINOBEL SMITH. *Art News*, **40** [17] 12 (1941); *Design*, **43** [4] 5, 19 (1941).—In the Latin American section of the Syracuse Museum's Western Hemisphere Exhibition are contemporary ceramics from 15 other nations of this hemisphere. Designs based on pre-Spanish decorations are well adapted to new techniques, especially the fine geometry of Brazilian primitives combined with modern functionalism. Marina Nuñdez del Prado of Bolivia demonstrates in the exhibit actual modeling, firing, and glazing. "Llamas," one of her sculpture groups, and "Indian Mother" by Luis Guzman Reyes of Chile are illustrated. See "Tenth . . . ," *Ceram. Abs.*, **21** [1] 5 (1942). M.E.P.

French and Italian pottery from the Rothschild collection. C. LOUISE AVERY. *Bull. Metropolitan Mus. Art*, **36** [11] 229–32 (1941).—The Museum has obtained nine pieces from the distinguished collection of Baron Gustave de Rothschild. There are five examples of Italian majolica, one of Medici porcelain, and three of French faience, chiefly of the 16th century. A lustered plate from Deruta represents the transition from the abstract designs of the earliest majolica to the pictorial style that developed after 1525. The other four majolica pieces are examples of the pictorial style, the designs being adapted from contemporary paintings and engravings with mythological and dramatic subjects. A plate with a central medallion picturing the death of King Saul is an example of the famous Medici porcelain made at Florence in the latter half of the 16th century. The shape is borrowed from the Near East. The soft, waxlike quality of the glaze and the charm and delicacy of execution give great distinction to this piece. The Museum owns two other examples of Medici porcelain. The examples of French faience comprise a pedestal salt having the warm coloring of Palissy ware, a figure of a hurdy-gurdy player standing on a pedestal, glazed similarly to the other piece and following the Palissy tradition, and a large pilgrim bottle inscribed under the base, "Nismes 1581." It displays the coat of arms of John Casimir of Bavaria, Count Palatine of Simmern (1543–1592), and of his wife, Elizabeth, daughter of Augustus I, Elector of Saxony. A similar bottle in the collection of J. P. Morgan and a dish in the Victoria and Albert Museum, London, are outstanding examples of the work of Antoine Sigalon (1524–1590), a Huguenot potter of Nîmes. A.A.A.

Greek accessions. CHRISTINE ALEXANDER. *Bull. Metropolitan Mus. Art*, **36** [10] 202–204 (1941).—A red-figured Athenian calyx krater depicting a scene in Circe's palace has recently been placed on exhibition. The pictures tell the story of Odysseus arriving at the palace to rescue his companions who have been transformed into animals by Circe. The krater is from the third quarter of the 5th century B.C., and its subject has not been previously represented in the Museum's collections. A.A.A.

Han dynasty tombs in Szechwan. DAVID CROCKETT GRAHAM. *Asia*, **42**, 59–63 (1942).—These tombs are either in caves or in separate structures of ornamented brick. Some designs simulate the wooden latticework of the period, while others include animals, men, geometric patterns, and written inscriptions. The objects placed in these tombs were of jade, bronze, iron, terra cotta, and earthenware and included vases, jugs, jewelry, animals, warriors, scholars, farmers, houses, and ornamented bell-shaped objects. Most of the pottery is of gray clay and unglazed, but some has a fine golden color. Some of the pieces were made on a wheel. A rare porcelanous ware with a greenish-brown glaze was also found. W.D.F.

Letter from Benjamin Franklin. ANON. *Jour. Roy. Soc. Arts*, **89** [4598] 735–36 (1941).—The Royal Society of Arts in England invited Benjamin Franklin to become a corresponding member of the Society in Sept., 1755, and he accepted the invitation as "a very great Honour" from Philadelphia in Nov., 1755. He enclosed 20 guineas "to be applied by the Society in premiums for some Improvements in Great Britain as a Grateful tho' small return for their most kind and Generous Intentions of Encouraging Improvements in America." In a later letter from London dated Nov. 3, 1773, to an unknown person (*ibid.*, October 13, 1941) Franklin thanked the recipient for "specimens so kindly sent of your new art of engraving. That on the china is admirable." He did not know who invented the copperplate engraving for earthenware but praised "the improvement in taking impressions not directly from the plate but from printed paper, applicable to other than flat forms." He referred modestly to his own proposals "sent from America to Dr. Mitchell, 20 years earlier, for the printing of square tile for ornamenting chimneys from copper plates, describing the manner in which it might be done." Dr. Mitchell communicated the scheme "to several artists in an earthen way about London who rejected it as impracticable." K.R.

Medieval stove ceramics in Lübeck. PIEPER. *Keram. Rundschau*, **48** [51] 431–33 (1940).—The discovery of a large number of tile and ceramic fragments of the late

Gothic period is described. They present mostly scenes in the life of Christ and other religious subjects. Illustrated. M.H.

Miniature pottery and its potting. MARGARET STEENROD FETZER. *Bull. Amer. Ceram. Soc.*, 21 [1] 12–14 (1942).—14 photos.

Herman Mueller. ANON. *Bull. Amer. Ceram. Soc.*, 21 [1] 1–3 (1942).—2 photos.

Portrait of a potter. ROLLO WALTER BROWN. *Harper's Mag.*, No. 1099, pp. 91–97 (1941).—This penetrating and revealing portrait of B.'s father shows him as an unusually interesting pioneer in Civil War times when people "were looking for something tight enough to 'keep' fruit and vegetables in." While some of the men were grubbing saplings from the hillsides they discovered clay. Little log potteries sprang up; they had a mill shed at one end, a potter's wheel or two and ware scaffolds and drying loft in the middle, a kiln and a fired-ware platform at the other end, and a ware yard out in front. If a man knew how to make even the crudest kind of ware or could engage someone to make it for him, he built one of these log potteries near his house, which might be in a hollow where clay and water were at hand or on a ridge where neither was to be had without especial effort. Anybody could load ware into a wagon and peddle it with more or less profit. "The ancient craft called to the young man, who did not like the constant hurt of wood smoke in his eyes. He apprenticed himself to a neighbor who was a good potter; he quickly became a potter in his own right in a pot shop his father had built 'down in the hollow'. Eventually he was living in a log house of his own, high on a ridge a distance away." After working in his garden every morning from 4:30 until breakfast, he walked to the pot shop "and kicked the wheel all day. At intervals he stopped the wheel, wiped the slushy clean-smelling clay from his hands, and sprinkled the clay the horse was grinding in the mill shed or took up the ground clay from the mill and put in a new grinding from the clay pen. Or he balanced a long board of drying ware on his hands and carried it out into the sun . . . " From time to time he set the kiln for an entire day and then stayed up all night to fire with huge 4-ft. sticks of dried oak. Some time the next day he "burned off" the kiln and left the ware to cool down slowly for several days; he then carried it to the ware yard. "There, piled in attractive symmetrical arrangements, were blue-gray or black fruit jars, jugs, water jars, Dutch pots, butter jars, milk crocks, and churns." K.R.

Two early Greek vases. G. M. A. RICHTER. *Bull. Metropolitan Mus. Art*, 36 [9] 187–90 (1941).—A Corinthian plate dating from about 600 to 575 B.C., decorated with a fine painting of a chimaera, and an Athenian amphora showing the contest of Herakles and the Nemean Lion, dating from about 550 B.C., have recently been purchased by the Museum. The plate is a fine example of Corinthian ware when Corinth was at the height of its prosperity and is no doubt the work of the Chimaera Painter, so named from his design on a plate in Vienna and a similar plate in the Louvre. The Athenian black-figured amphora is of the period when Corinthian ware was not so popular as that of Athens. The graceful shape, with its warm terra-cotta color and black glaze, is distinctively Athenian. The painting appears on both sides with slight variations. Although similar to the work of Exekias, this amphora has not yet been identified with any particular painter's work. A.A.A.

Wrinkled old woman is art from Peru's Indian past. EMILY C. DAVIS. *Science News Letter*, 40 [20] 310 (1941).—The wrinkled countenance of an old woman adorns a pottery vase which is one of the discoveries in a new-found chapter of Peru's prehistoric past. G.A.K.

SEPARATE PUBLICATION

Symposium on Color—Its Specification and Use in Evaluating the Appearance of Materials: Introduction to color. DEANE B. JUDD. Pp. 1–17. **Color specifications of transparent materials.** FRANCIS SCOFIELD. Pp. 18–22. **Hiding power and opacity.** ROSCOE H. SAWYER. Pp. 23–36. **Color standards for opaque materials.** I. H. GODLOVE. Pp. 37–46. **Spectrophotometry and color evaluation.** A. E. PARKER. Pp. 47–60. **Photoelectric tristimulus colorimetry.** RICHARD S. HUNTER. Pp. 61–79. Published by American Society for Testing Materials, Philadelphia, 1941. Price, paper cover, $1.00; cloth, $1.50. Sponsored jointly by the American Society for Testing Materials and the Inter-Society Color Council. R.A.H.

PATENTS

Designs for:

Bottle. F. R. HOCH (T. J. Holmes Co., Inc.). U. S. 130,920, Dec. 30, 1941 (Sept. 16, 1941).

Drinking glass. J. E. SPENCE (Hazel-Atlas Glass Co.). U. S. 130,857, Dec. 23, 1941 (Oct. 6, 1941).

Plate. P. L'ANGLAIS (Mayer China Co.). U. S. 130,939, Dec. 30, 1941 (Oct. 28, 1941). J. I. BAHL (Bahl Potteries, Inc.). U. S. 130,910, Dec. 30, 1941 (March 27, 1941).

Forming vitreous decalcomanias. M. A. CIAVOLA. U. S. 2,267,787, Dec. 30, 1941 (July 18, 1938).—The method of forming self-supporting decalcomanias containing vitrifiable material capable of being transferred to a vitreous surface and fired thereon to produce an opaque design which comprises forming directly upon decalcomania paper a film of material including pulverized glass and a binder which is decomposed during firing by squeegeeing a sufficient amount of the material forming the film through at least one silk-screen stencil onto the transfer paper to form the material into a self-supporting film when removed from the paper and to form the opaque design when applied to the surface and fired.

Cements

PATENT

Cement powders and method of producing acidproof mortars therefrom. KARL DIETZ AND FRANZ PRIVINSKY (Pen-Chlor, Inc.). U. S. 2,269,096, Jan. 6, 1942 (July 29, 1939).—A method of producing acidproof mortars which consists in mixing cement powders consisting of pulverulent aromatic sulfochlorides and acidproof filling substances with potassium silicate and water.

Enamel

All-weather highway—all-weather signs. C. D. CAMAHAN. *Ind. Gas*, 19 [9] 5 (1941).—Porcelain enameled signs for the Pennsylvania Turnpike were made from steel cleaned in gas immersion-fired cleaning tanks. S.S.

Behavior of sodium fluosilicate. VIELHABER. *Emailwaren-Ind.*, 17 [23–24] 65 (1940).—Sodium fluosilicate is the sodium salt of fluosilicic acid; for enamel technique it is composed of 0.446 NaF and 0.554 SiF_4. About one half of the SiF_4 volatilizes unless a substance (soda) is added to react with it; 7.35 parts sodium fluosilicate and 3.7 parts soda give the same fusibility and opacification as 6 parts sodium fluosilicate, and the enamel will not craze. See "Loss . . . ," *Ceram. Abs.*, 18 [5] 122 (1939). M.V.C.

Calcite in boron-free enamels. VIELHABER. *Emailwaren-Ind.*, 17 [17–18] 47 (1940).—Calcite is used in enamels as a flux. It has an unfavorable effect on the solubility of other constituents in the enamel slip, especially

alumina, which will separate out if large quantities are present. Fluorspar does not have this effect to such a marked degree. The more calcite is added, the less alumina should be present, and as the alumina-bearing constituent is usually the feldspar alone it should be reduced in quantity. As feldspar is a good preopacifier for white enamels, this substitution is made only in ground or colored enamels and glazes where opacification is not important.
M.V.C.

Effect of firing treatment on physical properties of dry-process enamels. PERRY C. MCCOLLOM AND R. R. DANIELSON. *Jour. Amer. Ceram. Soc.*, 25 [2] 48–50 (1942).—4 references.

Effect of set of enamels on hydrometer readings of the slip. H. L. COOK AND C. H. WOLFF. *Jour. Amer. Ceram. Soc.*, 25 [2] 51–52 (1942).

Feldspar-quartz ratio in enamels. VIELHABER. *Emailwaren-Ind.*, 17 [29–30] 77–78 (1940).—SiO_2 is used in enamels in amounts of 30 to 60%; it is introduced as quartz or other minerals containing SiO_2, preferably feldspar which melts easier than quartz and is a cheap source of alkalis. When quartz alone is used with fluxes, the melting point of the enamel is irregular and smelting is difficult; the interval between softening and melting, however, which is of great importance in the application of the cover enamel and influences gloss and adherence to the ground, is not as high as when feldspar and quartz are used together. The proportion of feldspar to quartz that gives the greatest softening interval is either 1:2 or 2:1. Feldspar equalizes the irregularity of the smelting temperature caused by the use of quartz alone and is also an opacifier for enamels; it should not exceed 50% of the batch, however, and clay should not be present. These factors do not apply for ground enamels for sheet metal and must be modified in the case of boron-free enamels.
M.V.C.

Gas gets a workout on stoves. H. H. SLAWSON. *Ind. Gas*, 20 [5] 3 (1941); see "Automatic . . . ," *Ceram. Abs.*, 20 [6] 139 (1941). S.S.

Pyrolusite. ANON. *Emailwaren-Ind.*, 17 [17–18] 47–48 (1940).—The principal use of pyrolusite in the enamel industry as an addition to ground enamels for sheet metal is not that of an adhering oxide, because its adhering action is very slight, but rather that of an oxidizing agent. It has the advantage of giving off oxygen more slowly than saltpeter. Pyrolusite opacifies enamels, makes them dense, and gives a uniform coloring. Saltpeter as an oxidizer in ground enamels holds the metal oxide in a higher oxidation form and burns out impurities; a uniform color may also be obtained by the addition of a metal oxide, iron oxide being the simplest. The enamel takes up as much as 20% iron oxide from the underlying sheet metal, thus giving an irregular coloring; the addition of up to 10% iron oxide to the melt is necessary to make the coloring uniform.
M.V.C.

Radiant-tube enameling. W. F. ROSS. *Ceram. Ind.*, 35 [3] 66 (1940); *Ind. Gas*, 19 [6] 3, 4 (1940).—A gas-fired furnace with recuperator-type heat exchanger and carbide radiant tubes for the enameling of flatware, pressed and drawn shapes, etc., has been installed at the plant of the Porcelain Metal Products Co., Salem, Ohio. See "Enameling . . . ," *Ceram. Abs.*, 19 [11] 253 (1940). S.S.

Utilization of porcelain enamel: I. EMERSON P. POSTE. *Machine Design*, 13 [10] 56–58 (1941).—P. first describes the physical and chemical properties of porcelain enamel. Tests with 100% citric acid and the classification of enamels derived from these tests are described. **II, Sheet-metal parts.** *Ibid.*, [11] 62–65, 126, 128.—Ingot iron and specially refined low-carbon steel can be used; both are products of the open-hearth furnace with very low C, S, and P contents. To prevent warpage, sagging, chipping, hairlining, etc., a sheet which is not too thin should be selected. Maximum areas are given for 24-, 22-, and 20-gauge sheet. Construction details are described. Enamels for use with foods should have contents below the following maxima: 0.025 gm. total solids per sq. in. wetted surface and 0.35 mgm. Pb, 1 mgm. Sb, and 1 mgm. F per 10 sq. in. of wetted surface. No lead compounds are used. The enamel is applied by dipping or spraying. To prevent sagging, the sheet must be supported. The enamel should be as thin as possible. To obtain 75% reflectance, opaque enamel must be 0.012 in. thick, and superopaque enamel 0.008 in. thick. Maxima for these enamels are set at 0.020 in. and 0.015 in., respectively, by the manufacturer. **III, Cast-iron parts.** *Ibid.*, [12] 77–79, 144, 146.—An average analysis for cast iron suitable for enameling is as follows: Si 2.50, Mn 0.70, P 0.75, S 0.075, total C 3.30 to 3.40, combined C 0.50 to 0.60, and graphite 2.80 to 2.90%. Si should be lower for thicker sections and higher for thinner sections. Another helpful formula is as follows: (%Si+%P)/3 + C(total) = 4.3%. The structure should be uniform and dense. Design details are described which aid in obtaining uniformity and in preventing warping and hairlining. The condition of the molding sand (moisture) is important. Graphitic facings should be avoided. The enamel now preferred is the hard grade resistant to all acids except HF (no Pb is used). Soft enamels are easier to apply and are sometimes preferred. Both the dry and wet processes are being used. They control the design to a large degree (gravity deposition, applicability of rotary tables, etc.). Dry-process enamels are several times thicker than wet-process enamels, both being thicker than enamels applied to sheet metal. See "Chemistry . . . ," *Ceram. Abs.*, 20 [1] 7 (1941). J.M.N.

PATENTS

Making sinks, washbasins, etc. JACQUES STANITZ (Steel Plumbing Wares, Inc.). U. S. 21,997, Jan. 6, 1942 (Dec. 18, 1940); reissue of original U. S. 1,916,910, July 4, 1933 (Dec. 7, 1932).

Making white clouded enamel. IGNAZ KREIDL. U. S. 2,267,928, Dec. 30, 1941 (Aug. 21, 1936; Jan. 29, 1941).—In a method of producing a gas-clouded vitreous enamel, the step of incorporating in the enamel batch a compound containing cerium in an amount less than 1% of the enamel.

Tub construction. C. H. WIDMAN (Murray Corp. of America). U. S. 2,269,748, Jan. 13, 1942 (Oct. 19, 1939).

Glass

Analysis of gases dissolved in glass and gases present in seeds. HUGH A. SHADDUCK AND ARTHUR VAN ZEE. *Jour. Amer. Ceram. Soc.*, 25 [3] 69–83 (1942).—19 references, 12 figures.

Decrepitude of old bottles: aging, weathering, and solarization compared with use and abuse. L. G. GHERING AND F. W. PRESTON. *Jour. Amer. Ceram. Soc.*, 25 [2] 53–55 (1942).—6 references.

Dielectric strength of glass—an engineering viewpoint. E. B. SHAND. *Elec. Eng.*, 60 [8] 814–18 (1941).—The purpose of this paper is to provide the engineer with coordinated data and information regarding the dielectric breakdown of glass which will permit him to apply this insulating material more effectively for high dielectric strength. Particular attention is given to "edge effects" and other factors which cause wide variations in breakdown voltages. The data curves included refer mainly to Pyrex brand glass, but corresponding data have also been included on electrical porcelain because of the interesting comparisons shown. R.L.G.

Effect of barium oxide and zinc oxide on properties of soda-dolomite lime-silica glass. OWENS-ILLINOIS GLASS CO. GENERAL RESEARCH LABORATORY. *Jour. Amer. Ceram. Soc.*, 25 [3] 61–69 (1942).—8 references, 19 figures.

Glass-batch mixing. WAYNE E. BROWNELL AND S. R. SCHOLES. *Jour. Amer. Ceram. Soc.*, 25 [2] 56–59 (1942).—10 figures.

Glass industry—output expanded to meet demand and rearmament. ANON. *Index*, 21 [4] 88–94 (1941).—A brief review is given of the development of the glass industry

and the comparative value of the industry's annual output on the basis of the U. S. Census of Manufacturers for 1939. Because of the war, imports have been considerably curtailed while exports have been greatly expanded. The latest developments within the glass industry have resulted in the opening of new markets. An especially marked increase occurred in the output of glass building materials, textiles, containers, and tableware (increase of production of approximately 20% over 1940), plate and sheet glass, and special glass, particularly optical glass and glass for rearmament, such as glass for building up stereoscopic images from aerial photographs, etc. M.V.C.

Glass for mechanical parts. ANON. *Product Eng.*, 12 [11] 617–18 (1941).—Data on glass compositions taken from "Glass: The Miracle Maker" (*Ceram. Abs.*, 21 [1] 11 (1942)) are presented. M.H.

Glass is mostly gas, X-ray studies reveal. ANON. *Science News Letter*, 40 [21] 329 (1941). G.A.K.

Glass piping for ozone. ANON. *Ind. Chemist*, 16, 97–98 (1940).—Glass pipe manufactured by J. W. Towers & Co., Ltd., is used to convey ozone for sterilizing water in a swimming pool. The powerful oxidizing action of ozone is preserved by the glass piping accompanied by ebonite fittings. Illustrated. H.E.S.

Glass: today and tomorrow. ALEXANDER SILVERMAN. *Jour. Franklin Inst.*, 232, 239–49 (1941).—S. reviews recent advances in the use of glass and predicts future uses. W.D.F.

Glassmaking: I–X. B. M. PEARSON. *Ind. Chemist*, 16 [190] 314–18; [191] 352–56 (1940); 17 [192] 18–21; [193] 37–40; [195] 96–97; [196] 123–26; [197] 153–55; [198] 180–85; [199] 201–203; [200] 223–24 (1941).—P. presents a general review of the glass industry, covering the container or glass bottle industry, the window and plate glass industry, and the tableware or decorative glass field. Raw materials and their function and use are explained. The method of manufacture involving the use of highly mechanized equipment is shown, together with the important properties of glass, such as strength, viscosity, ductility, surface tension, etc. The preparation of the batch and the melting technique, refractories for the tanks, insulation, fuel technology, combustion control in the furnace, automatic bottle machinery, and annealing and sheet-drawing processes are also discussed. H.E.S.

History of circular dials of glass. H. LÖSCHNER. *Z. Instrumentenk.*, 60 [9] 281–83; [12] 371 (1940).—Calibrated dials of glass instead of metals for precision instruments, especially for angular measurements, were apparently first used in 1884 but did not find general use. They are advantageous in that glass is much less sensitive to atmospheric influences than metal, and the diamond can cut much finer, more uniform, and straighter lines on glass. Furthermore, the readibility in transmitted light is better and less fatiguing to the eye. The mounting of glass dials is briefly discussed. M.H.

Influence of temperature conditions on the streaking of plate glass. A. P. PATENKO. *Stekolnaya Prom.*, 14 [11] 5 (1938).—The basic cause of the formation of streaks is the heterogeneous distribution of temperature in the preheating chambers of the Fourcault channel. P. found that the higher the temperature of the glass mass in the preheating chamber, the lower is the streakiness of plate glass. M.V.C.

Modern furnace design and operation. S. R. SCHOLES. Presented at Conference on Glass Problems, Univ. of Illinois, Nov., 1941; abstracted in *Glass Ind.*, 22 [12] 516 (1941). A.P.

New process for glass as an art medium. THEODORE LAYMON. *Glass Ind.*, 22 [12] 518–20 (1941).—The technique of Joep Nicolas is based upon the use of flashed opaque glass with a fired-in pattern and partially etched backgrounds. See "Stained . . . ," *Ceram. Abs.*, 20 [8] 192 (1941); "Nicolas . . . ," *ibid.*, 21 [1] 4 (1942). A.P.

Recent research on glass in Department of Ceramic Engineering. C. W. PARMELEE AND A. E. BADGER. Presented at Conference on Glass Problems, Univ. of Illinois, Nov., 1941; abstracted in *Glass Ind.*, 22 [12] 515 (1941). A.P.

Recent work on cords. W. B. SILVERMAN. Presented at Conference on Glass Problems, Univ. of Illinois, Nov., 1941; abstracted in *Glass Ind.*, 22 [12] 515 (1941). A.P.

Report of progress in the metallurgical study of cast iron for glass molds. W. H. BRUCKNER AND H. CZYZEWSKI. Presented at Conference on Glass Problems, Univ. of Illinois, Nov., 1941; abstracted in *Glass Ind.*, 22 [12] 516 (1941). A.P.

Sources of stones in glass. D. W. ROSS. Presented at Conference on Glass Problems, Univ. of Illinois, Nov., 1941; abstracted in *Glass Ind.*, 22 [12] 516 (1941). A.P.

Standardization of laboratory glassware. FRITZ HILDEBRANDT. *Chem. Fabrik*, 12 [27–28] 335–36 (1939).—The use of German standards in Japan is described. J.M.N.

Systematic construction tables for thin cemented aplanatic lenses. C. D. BROWN AND T. SMITH. *Proc. Roy. Soc.* [London], **A178**, 525 (1941).—The tables give the information needed for the construction of aplanatic lenses made of only two refracting media, when the object is at infinity and when the image is equal in size to the object. A.P.

Water-hammer breakage in glass containers. T. C. BAKER. *Glass Ind.*, 22 [10] 430–33; [11] 469–74, 485–86; [12] 521–24, 534–36 (1941).—Water-hammer breakage of glass containers may occur whenever a heavy object is dropped a few feet upon a packed carton placed to permit a short downward movement of the jars. This occurs when one carton is dropped upon another in loading and stacking. The sudden acceleration forms cavities between the contents and the bottom of the jars in the lower carton and finally causes the contents to overtake the bottoms with a sharp impact. This impact consists of (1) a moderate hydrostatic pressure of 35 to 250 lb. per sq. in. which is at a maximum at the bottom and lasts for $^1/_{10,000}$ of a second and (2) an intense local pressure of 50,000 to 500,000 lb. per sq. in., lasting only a few microseconds, which is developed by the high velocity of the water at the point where the cavities collapse. Combinations of these two phenomena produce nine characteristic fracture patterns. Accurate tests on water-hammer strength of jars are difficult because the testing machine, the nature and location of adventitious flaws in the glass, and the nature of the contents influence the results. Breakage may be reduced by (1) prevention of common flaws in the glass, (2) more careful handling of cartons, (3) proper design of bottle and carton, (4) upside-down packing, and (5) moderate changes in the consistency of liquid contents or of headspace pressure. A.P.

X-ray diffraction and physical properties of potassium borate glasses. ROBERT L. GREEN. *Jour. Amer. Ceram. Soc.*, 25 [3] 83–89 (1942).—11 references, 10 figures.

PATENTS

Apparatus for firing fluorescent lamp tubing. R. M. GARDNER (Hygrade Sylvania Corp.). U. S. 2,267,727, Dec. 30, 1941 (Jan. 27, 1940).—A machine for firing internally coated tubes of glass, etc., the machine comprising an oven, a series of spaced refractory rollers along the bottom of the oven, a series of refractory forks, with each fork in the space between successive rollers, means for raising the forks, and a series of burners between the top of the oven and the rollers to direct heat downwardly toward the rollers.

Apparatus for handling sheet glass. J. L. DRAKE (Libbey-Owens-Ford Glass Co.). U. S. 2,267,605, Dec. 23, 1941 (Jan. 9, 1939).

Apparatus for making glass tubes. G. P. DESPRET (Compagnies Réunies des Glaces et Verres Spéciaux du Nord de la France). U. S. 2,267,554, Dec. 23, 1941 (Nov. 17, 1939).

Apparatus for production of glass threads. WILHELM ESSER (Oscar Gossler Glasgespinst-Fabrik G.m.b.H.). U. S. 2,267,019, Dec. 23, 1941 (Oct. 15, 1937).

Apparatus for toughening glass. PILKINGTON BROS., LTD. Brit. 541,554, Dec. 17, 1941 (July 13, 1939).

Building tile containing cellular glass. E. H. HAUX (Pittsburgh Plate Glass Co.). U. S. 2,268,251, Dec. 30, 1941 (May 20, 1938).—A building block comprising a fired clay shell and an expanded cellular glass body filling the inside of the shell and joined to the walls thereof and forming with the latter an integral composite unit.

Ceramic marble. ALEX VIDA. U. S. 2,266,926, Dec. 23, 1941 (Sept. 19, 1939).—The method of producing ceramic marble by means of a glass plate which comprises the steps of applying strands upon the face of the plate, applying a coat of ceramic color to the plate, the coat being composed of a mixture of powdered colored glass and a liquid vehicle, allowing the coat to partially dry and then removing the strands whereby channels are formed in the coat, applying a second coat of powdered colored glass mixed with a liquid vehicle over the first coat to fill the channels, the partially dried color of the first coat adjacent to the channels blending with the portion of the color of the second coat received in the channels to form colored veins whose edges blend into the color of the first coat, permitting the second coat to dry, and fusing the powdered colored glass onto the glass plate for causing it to become a permanent part thereof.

Ceramic-to-metal seals. BRITISH THOMSON-HOUSTON Co., LTD. Brit. 540,991, Nov. 19, 1941 (July 14, 1939).

Drive for glassforming machines. WALTER WINN. U. S. 2,266,838, Dec. 23, 1941 (Feb. 28, 1938).

Electric furnaces suitable for glassmelting. BRITISH THOMSON-HOUSTON Co., LTD. (J. Ferguson). Brit. 541,033, Nov. 19, 1941 (Jan. 31, 1941).

Glass article separating method. G. L. CURRY AND A. N. SMITH (Corning Glass Works). Can. 401,490, Dec. 16, 1941 (March 27, 1940; in U. S. April 14, 1939). G.M.H.

Glass article shaping apparatus. WALTER C. WEBER (Corning Glass Works). Can. 401,491, Dec. 16, 1941 (May 8, 1940; in U. S. June 7, 1939). G.M.H.

Glass charging and pressing apparatus. D. E. GRAY (Corning Glass Works). Can. 401,486, Dec. 16, 1941 (March 1, 1939; in U. S. March 3, 1938). G.M.H.

Glass furnace. S. A. FORTER (Forter-Teichmann Co.). U. S. 2,268,546, Jan. 6, 1942 (Oct. 6, 1938).

Glass furnaces. W. E. EVANS (O'Neill European Machine Co. of Montreal, Canada). Brit. 541,111, Nov. 26, 1941 (May 7, 1940).

Glass machine and method of making glass. GEORGE ROESSLER. U. S. 2,269,553, Jan. 13, 1942 (Oct. 8, 1936).

Glass-pressing apparatus. L. F. GOODRICH (Corning Glass Works). U. S. 2,267,236, Dec. 23, 1941 (Aug. 30, 1938).

Glass strand coating apparatus. EDWIN J. FISHER (Fiberglas Canada, Ltd.). Can. 400,986, Nov. 25, 1941 (July 31, 1939). G.M.H.

Glass-tank wall and method of construction. G. J. EASTER (Carborundum Co.). U. S. 2,269,445, Jan. 13, 1942 (May 14, 1938).

Glass-tempering bath. RALPH K. DAY (Corning Glass Works). Can. 401,488, Dec. 16, 1941 (June 13, 1939; in U. S. June 22, 1938). G.M.H.

Glassware-blowing mechanism for forming hollow glass articles. E. G. BRIDGES. Brit. 541,440, Dec. 10, 1941 (Sept. 2, 1940).

Glassware lehr. D. G. MERRILL (Hartford-Empire Co.). U. S. 2,268,609, Jan. 6, 1942 (July 8, 1939).

Glassworking machine. J. W. JUVINALL AND W. B. WINES (Western Electric Co., Inc.). U. S. 2,267,108, Dec. 23, 1941 (Oct. 4, 1939).—In a glassworking machine, complementary mold members cooperating to provide a mold cavity, cooperating clamping jaws for centering and holding a glass tube within the cavity, means for closing the mold members, and means rendered effective by the closing of the mold members for closing the clamping members.

Glass-yarn producing apparatus. PIERO MODIGLIANI (Fiberglas Canada, Ltd.). Can. 400,985, Nov. 25, 1941 (Oct. 28, 1938). G.M.H.

Heat-resisting mirror. R. E. RICHARDSON (Duplate Safety Glass Co. of Canada, Ltd.). U. S. 2,268,168, Dec. 30, 1941 (Nov. 21, 1939).—The method of making a heat-resisting mirror which comprises forming an aluminum reflecting surface on glass having a low coefficient of expansion, coating the reflecting surface with a mixture of particles of vitreous enamel having a coefficient of expansion higher than that of the glass but fusible at a temperature low enough to avoid injury to the reflecting surface and which when vitrified is substantially inert to aluminum and of particles of quartz of a size at least below 200-mesh having a coefficient of expansion lower than that of the glass but infusible at or near the temperature of fusion of the first-mentioned material and substantially inert to aluminum, and subjecting the glass so coated to a temperature sufficient to cause fusion of the first-mentioned material.

Hermetic sealing of metal into vitreous materials. SIEMENS ELECTRIC LAMPS & SUPPLIES, LTD., J. N. ALDINGTON, AND A. J. MEADOWCROFT. Brit. 541,208, Dec. 3, 1941 (Feb. 15, 1940).

Insulator press. WALTER C. WEBER (Corning Glass Works). Can. 401,487, Dec. 16, 1941 (March 1, 1939; in U. S. March 19, 1938). G.M.H.

Laminated safety glass and process of producing. J. D. RYAN (Libbey-Owens-Ford Glass Co.). U. S. 2,268,266, Dec. 30, 1941 (March 12, 1938).

Machine for molding glass articles. R. G. ALLEN (Owens-Illinois Glass Co.). U. S. 2,269,391, Jan. 6, 1942 (April 7, 1939).

Making colored sheet glass. J. H. FOX (Pittsburgh Plate Glass Co.). U. S. 2,268,247, Dec. 30, 1941 (Dec. 15, 1937).

Manufacturing stemmed glassware. A. J. CUNNINGHAM (A. H. Sharpe). U. S. 2,269,010, Jan. 6, 1942 (Oct. 20, 1939).

Metal-to-glass seals. GENERAL ELECTRIC Co., LTD., R. L. BREADNER, AND R. W. DOUGLAS. Brit. 540,742, Nov. 12, 1941 (April 26, 1940). STANDARD TELEPHONES & CABLES, LTD., AND C. STRACHEY. Brit. 541,381, Dec. 10, 1941 (May 24, 1940).

Method and apparatus for the continuous manufacture of tubes of glass. COMPAGNIES RÉUNIES DES GLACES ET VERRES SPÉCIAUX DU NORD DE LA FRANCE. Brit. 540,759, Nov. 12, 1941 (Nov. 19, 1938).

Method and apparatus for toughening sheets of glass. B.I.G. SECURITIES, LTD. (R. Gajoti). Brit. 541,049, Nov. 26, 1941 (May 8, 1940).

Optical glass making apparatus. J. C. HOSTETTER (Corning Glass Works). Can. 401,489, Dec. 16, 1941 (Jan. 12, 1939; in U. S. Feb. 7, 1939). G.M.H.

Preparing finely comminuted oxides. DAN MCLACHLAN, JR. (Corning Glass Works). U. S. 2,269,059, Jan. 6, 1942 (May 14, 1938).—The method of preparing finely divided oxides which includes dissolving a hydrolyzable compound in a nonaqueous liquid medium comprising at least one liquid in which water is substantially insoluble, the concentration of the compound per 500 cc. of solvent being not less than the equivalent of 1 mol of the oxide, introducing into the solution slowly and with agitation an amount of water which is substantially the stoichiometric quantity necessary to convert the compound to the oxide, the concentration of the water being not less than that of the compound, and thereafter separating the oxide by filtration.

Producing vitreous silica articles. J. A. HEANY (Heany Industrial Ceramic Corp.). U. S. 2,268,589, Jan. 6, 1942 (July 7, 1934).—A method of producing vitreous, translucent, nonporous silica articles of high strength and density which comprises forming and firing an article composed of a reactive, readily vitrifying, finely divided silica obtained by the decomposition of gaseous silicon compounds, the firing being carried out at a temperature above 1200°C. but below the fusion point of silica.

Production of safety glass. A. TILLYER AND BRITISH INDESTRUCTO GLASS, LTD. Brit. 540,734, Nov. 12, 1941 (April 25, 1940).

Reflector and process of manufacture. W. O. LYTLE (Pittsburgh Plate Glass Co.). U. S. 2,268,258, Dec. 30, 1941 (July 15, 1939).—A process of manufacturing a reflector which comprises forming a suitable legend in a vitreous enamel on the inner smooth surface of a tempered

glass plate provided with an outer figured surface, heating the plate sufficiently to fuse the vitreous enamel, applying a metallic reflecting film to the smooth surface of the tempered glass plate in and about the legend, adhering a backing member to the coated surface of the glass plate through the medium of an intermediate reinforcing layer of synthetic resin, and subsequently fracturing the tempered glass plate.

Rock or mineral wool manufacture. C. L. SAVIDGE (Ohio Insulation Co.). U. S. 2,269,231, Jan. 6, 1942 (March 8, 1937).

Sealing glass and ceramic material together. EDMUND LÖPP (C. Lorenz A.-G.). U. S. 2,268,666, Jan. 6, 1942 (Jan. 27, 1939).

Sheet glass forming machine. J. L. DRAKE AND G. R. ROESCH (Libbey-Owens-Ford Glass Co.). U. S. 2,267,604, Dec. 23, 1941 (July 17, 1937).

Take-out for glassware-forming machines. G. B. LANGER (Lynch Corp.). U. S. 2,268,075, Dec. 30, 1941 (Sept. 24, 1938).

Tempered glass article. J. B. MITFORD (American Securit Co.). U. S. 2,269,597, Jan. 13, 1942 (Jan. 4, 1937).

Tempering hollow glass articles. J. B. MITFORD (Corning Glass Works). U. S. 2,269,000, Jan. 6, 1942 (Nov. 2, 1939).

Translucent insulating building block. L. W. MULFORD. U. S. 2,269,123, Jan. 6, 1942 (March 21, 1938).

Uniting laminas to form laminated glass. ADOLF KÄMPFER. U. S. 2,268,489, Dec. 30, 1941 (Dec. 12, 1938).

Structural Clay Products

Selecting proper bonds in brickwork. ANON: *Amer. Builder*, **63** [8] 72–73, 114–16 (1941).—The effect of different bonds on the strength, appearance, and cost of brick walls is discussed, and the different bonds are classified by the relation between headers and stretchers and by the methods of breaking the joints. S.S.

SEPARATE PUBLICATIONS

Dimensions of Building Brick. British Standard Specification No. 657—1941. British Standards Inst., London. Price 1s.—The earlier standard specification has been extended to include all building brick. Such brick shall be $8^3/_4$ in. long, $4^3/_{16}$ in. wide, and either $2^5/_8$ in. or $2^7/_8$ in. thick. Where costly modifications in a brickmaking plant are necessary to provide brick of the sizes specified, the existing sizes may be continued until the needed alterations have been made. Apart from this, conformity to the standard dimensions becomes operative immediately. The specification does not apply to brick made in Scotland. A.B.S.

Effect of Outdoor Exposure on the Water Permeability of Masonry Walls. CYRUS C. FISHBURN, DOUGLAS E. PARSONS, AND PERRY H. PETERSEN. *Nat. Bur. Standards Bldg. Materials and Structures Rept.*, BMS76, 19 pp. (Aug., 1941). Supt. of Documents, Govt. Printing Office, Washington, D. C. Price 15¢.—The water permeabilities of about 100 small masonry wall specimens were measured before and after exposure to the weather at Washington, D. C., for a maximum period of 3 years. Some of the more permeable walls had been waterproofed before exposure, and the durability of the waterproofing treatments, as well as the structural appearance and condition of the walls, was also determined. The exposure did not have an important effect on the permeability of all-brick or brick-faced walls 8 in. or more in thickness. The permeability of stucco-faced walls was slightly increased. Repointing of the face joints was the most effective and durable waterproofing treatment. R.A.H.

Strength, Absorption, and Resistance to Laboratory Freezing and Thawing of Building Brick Produced in the United States. JOHN W. MCBURNEY AND JOSEPH C. RICHMOND. *Nat. Bur. Standards Bldg. Materials and Structures Rept.*, BMS60, 56 pp. (Nov., 1940). Supt. of Documents, Govt. Printing Office, Washington, D. C. Price 15¢.—This report gives the compressive and transverse strengths, water absorptions, saturation coefficients, and either the number of cycles of freezing and thawing required to produce failure or the loss in weight after 51 cycles for 3368 building brick representing 525 gradings produced by 209 manufacturers. Average results for all tests except freezing and thawing are given for an additional 197 gradings or samples comprising a total of 1190 specimens. These 197 gradings include samples from 20 additional manufacturers. R.A.H.

PATENTS

Glazed building block. E. S. GUIGNON, JR. U. S. 2,269,018, Jan. 6, 1942 (Feb. 19, 1940).

Treating clays. G. J. BARKER AND EMIL TRUOG (Wisconsin Alumni Research Foundation). U. S. 2,268,131, Dec. 30, 1941 (April 6, 1938).—In the manufacture of brick, etc., formed by the stiff-mud process from a clay mass sufficiently stiff so that it does not run under the influence of gravity, that improvement which comprises incorporating in the clay mass a basic material from the group consisting of the hydroxides and carbonates of the alkali metals and ammonia in an amount such that at least 10% of the base-exchange capacity of the clay mass is saturated with basic constituents from the group consisting of the alkali metals and ammonia and further limiting the quantity of the basic material incorporated in the clay mass to an amount which gives the clay mass a pH within the range 6 to 8.5 for acid clays and within the range 7.3 to 9.5 for nonacid clays.

Refractories

Aluminum alloys—their possibilities of future development. EINERL AND NEURATH. *Chem. Age* [London], **45** [1166] 237–39 (1941).—The raw material used for the production of aluminum consists almost entirely of bauxite (containing about 55 to 65% Al_2O_3, approximately 28% Fe_2O_3, up to 4% SiO_2, and 12 to 30% water); about 4 tons are necessary to produce 1 ton of aluminum. The world production of bauxite in 1938 was as follows: France, 680,000; Italy, 380,000; Germany, 90,000; Balkan states, 1,000,000; Russia, 250,000; the U. S., 430,000; and British and Dutch Guiana and the Dutch East Indies, 1,000,000 tons. About two thirds of the bauxite mined is utilized for making aluminum for refractories and alumina cement; the remaining third is used by the oil-refining and chemical industries. A.B.S.

Aluminum from western alunites. FRANK EICHELBERGER. *Mining Congress Jour.*, **27** [11] 37–39 (1941).—The Kalunite process is adapted to treat not only alunite but also high-alumina clays and high-silica bauxites. The alunite is dehydrated and treated with H_2SO_4 and K_2SO_4 to form potash alum. This step is for purification. The potash alum solution is autoclaved at 200°C. and 250 lb. per sq. in. to give a basic potassium aluminum sulfate analogous to alunite. This sulfate is ignited to drive off sulfur oxides, and the K_2SO_4 is then leached out with water. Aluminum produced from this alumina compares well with other aluminum. High-alumina clays may be the future source of aluminum. See "New . . . ," *Ceram. Abs.*, **21** [1] 15 (1942). W.D.F.

Coke-oven refractories. W. C. RUECKEL. *Amer. Gas Assn. Proc.*, **21**, 567–72 (1939); reprinted in *Refrac. Jour.*, **17** [7] 239–52 (1941).—Silica brick allows higher coking rates because higher flue temperatures can be employed and its conductivity is higher than fire-clay brick. The

rate of expansion of silica brick is high at low temperatures and practically zero at high temperatures. Silica brick has a greater high-temperature load-bearing strength. Oven failures occur because of the use of expanding coal, settlement of foundations, growth of brick in service, reaction between ash in coal and oven lining, poor operating practices, and expansion stresses due to variations in temperature. See *Jour. Amer. Ceram. Soc.*, 21 [10] 354–60 (1938). S.S.

Forging furnaces for commercial production work. WALDEMAR NAUJOKS. *Heat Treating & Forging*, 27 [5] 222 (1941).—Standard firebrick shapes for forging furnaces are listed. The hearth should have 2½ in. of insulating firebrick on steel plate with 4 to 6 in. of rammed plastic magnesite or chrome plastic. The walls should be at least 9 in. thick, preferably 13½ in. The outer 4½ in. should be insulating firebrick tied with firebrick headers every fourth or fifth course. The inner lining should be superduty fire-clay brick laid with high-temperature cement. Arches may be plastic firebrick or regular firebrick, 6¼ to 9 in. thick; they may be of the ring, bonded, or bung type. The hearth piers should be superduty firebrick or superrefractories. Fuel selection depends largely on the cost of different fuels. Systematic inspection reduces the need for major repairs. The temperature head between the furnace and the metal varies with the metal being heated. General suggestions are given for furnace construction. See "Stainless . . ., "*Ceram. Abs.*, 18 [8] 218 (1939). S.S.

Knowledge of the slag as basis of steel metallurgy. FRIEDRICH KÖRBER AND WILLY OELSEN. *Stahl & Eisen*, 60 [42] 921–29; [43] 948–55 (1940).—The importance of phase diagrams is emphasized, and ternary diagrams are given, including $CaO–SiO_2–Al_2O_3$, $CaO–MgO–SiO_2$ (both at several temperatures), $CaO–SiO_2 + Al_2O_3$, FeO, CaF_2, or MgO at 1600°C., and $FeO–CaO–P_2O_5$ at 1600°C. Examples for evaluating the data for the steel process are given. J.M.N.

Making aluminum from clay. ANON. *Science News Letter*, 40 [21] 332 (1941).—In a new process for the manufacture of aluminum from clay, selected high-silica clays are used. The clay is digested with hydrochloric acid, and the resulting product is decomposed to obtain aluminum oxide. G.A.K.

Refractory mortars for coke ovens. V. A. BRON. *Ogneupory*, 8 [5–6] 257–64 (1940).—Mortars of sufficient binding power for silica linings in all zones of coke ovens were manufactured from Bilimbaevskiĭ quartzites. The mortars are mixtures of powdered Bilimbaevskiĭ quartzites, Troibanovskiĭ clay, and fired silica with or without the admixture of dustlike quartz, chalk, and soluble glass, depending on the coke-oven zone in which the mortars are to be used. M.V.C.

Sagger mixes from Ural raw materials. R. I. ROZENBERG. *Keram. Sbornik*, No. 14, pp. 21–25 (1941).—Experiments in the production of sagger mixes from different raw materials found in the Urals are described. See "Selecting . . .," *Ceram. Abs.*, 20 [9] 217 (1941). M.V.C.

Selecting refractories for gas-fired industrial furnaces. J. J. OFFUTT. *Heat Treating & Forging*, 27 [7] 351 (1941).—A first-quality firebrick is satisfactory for the major portion of most furnace linings. Special grades are used in areas where unusually severe conditions exist. Superduty brick is used for high temperatures, special hard-fired superduty brick for high temperatures and abrasive action, and high-alumina brick for higher temperatures. Insulating firebrick can be used in direct contact with the flame. The effect of furnace atmosphere on refractories must be considered. S.S.

Zirconia–magnesia spinel system. R. F. MATHER. *Jour. Amer. Ceram. Soc.*, 25 [3] 93–96 (1942).—8 references, 4 figures.

SEPARATE PUBLICATION

Blast-Furnace Cement. British Standard Specification No. 146—1941. British Standards Inst., London. Price 2s net.—This is the third revision of the standard specification, first issued in 1923. The cement consists of a mixture of Portland cement, clinker, and granulated blast-furnace slag in any desired proportions but not exceeding 65% of slag. The following tests are included: (*a*) *Fineness:* The residue on a British Standard Sieve No. 170 shall not exceed 10%. (*b*) *Composition:* The composition of the cement clinker present shall conform to that of the current British Standard Specification for Portland cement. The blast-furnace cement shall show less than 1% insoluble residue, less than 5% magnesia, less than 2% sulfuric anhydride, less than 1.2% sulfur present as sulfide, and less than 3% loss on ignition. (*c*) *Strength:* The ultimate tensile stress of a mixture of 1 part of cement and 3 parts, by weight, of standard sand, gauged with a suitable proportion of water, after 3 days and 7 days shall not be less than 300 lb. per sq. in. and 375 lb. per sq. in., respectively. The compressive strength test may, at the request of the purchaser, be substituted for the tensile stress test. In that case, the compressive strength of 2.78-in. cubes of the cement-sand mixture with exactly 10% of water shall not be less than 1600 lb. per sq. in. after 3 days or less than 2500 lb. after 7 days. The paste used and the cubes made from it must be molded, vibrated, and stored under conditions which are described in detail in the specification. (*d*) *Setting time:* The initial setting time, when tested with a Vicat apparatus, shall not be less than 30 min., and the final setting time shall be not more than 10 hr. For quick-setting cements, the initial setting time shall not be less than 5 min. and the final setting time more than 30 min. (*e*) *Soundness:* This is tested by the Le Châtelier method, as before. A.B.S.

PATENTS

Coated refractory melting pot. R. E. BOOTH AND JOSEPH SCORZONI (General Electric Co.). U. S. 2,269,176, Jan. 6, 1942 (June 26, 1940).—A container suitable for use with molten aluminum, etc., comprising a carbon-bonded silicon carbide receptacle having a vitreous coating on the outside surfaces which are in contact with the surrounding atmosphere during the heating thereof, the coating being capable of effectively protecting the receptacle from oxidation thereof and consisting of 40 to 60 parts by weight of flint and about 57 parts by weight of a fused inorganic binder comprising a fused mixture of an inorganic enamel, ball clay, borax, soda ash, and tin oxide.

Ingot-mold lining. GERHARD BEHRENDT (August Thyssen Hütte A.-G.). U. S. 2,266,734, Dec. 23, 1941 (April 21, 1939).—An ingot-mold lining for casting heavy steel ingots suitable for repeated use, including a thin lining body of refractory material, the material being composed of a mixture of grains of fired chamotte, fresh clay, and quartz with different sizes sintered to a high strength to resist the mechanical action of the casting material, the expansion coefficient of the different composite parts of the refractory material behaving oppositely under heat so that expansion and contraction do not occur during drying and casting.

Refractory. N. P. PITT, A. C. HALFERDAHL, AND F. E. LATHE (Canadian Refractories, Ltd.). U. S. 2,270,220, Jan. 13, 1942 (April 1, 1937).—A method of producing an improved refractory from natural chrome ore which comprises mixing with the chrome ore lime sufficient substantially only to convert free and combined silica in the mixture into dicalcium silicate and firing the mixture at a temperature in excess of 2600°F.

Refractory brickwork. W. R. MCLAIN. U. S. 2,266,785, Dec. 23, 1941 (Aug. 10, 1939).—In a refractory brickwork, a pair of opposed rows of contacting substantially identical rectangular brick similarly disposed in alignment, whereby the surfaces of the brick of each row are substantially flush, a similarly formed and disposed pair of opposed rows of brick in contact with but staggered with respect to the first-named pair of rows, the contacting faces of the brick of each row having opposed rectangular recesses, thereby providing a keyway between four brick forming a pair of rows and extending at right angles to the rows, and a rectangular key disposed in the keyway.

Refractory castings for glassmaking and other apparatus. R. HADDAN (Corning Glass Works). Brit. 541,544, Dec. 10, 1941 (March 27, 1941).

Refractory and method of making. EUGENE WAINER AND D. S. HAKE (Titanium Alloy Mfg. Co.). U. S. 2,267,772, Dec. 30, 1941 (July 20, 1939).—A refractory composition comprising zircon, a minor quantity of a fluosilicate, and a minor quantity of a material taken from the class consisting of phosphoric acid and zirconium dioxide.

Refractory nozzle. W. S. DEBENHAM AND W. R. MCLAIN. U. S. 2,268,279, Dec. 30, 1941 (Sept. 12, 1939).

Solubilizing claylike minerals. KARL BÜCHE AND HANS GINSBERG (Th. Goldschmidt Corp.). U. S. 2,267,490, Dec. 23, 1941 (Oct. 31, 1938).—In the extraction of alumina from clay with acid, the process which comprises forming a mass of siliceous raw clay into coarse granules of substantially uniform size not substantially less than 5 mm. in diameter, calcining the granules at a temperature ranging from about 600° to 800°C. under conditions of direct heating, whereby the cores of the granules are rendered acid soluble but acid-insoluble incrustations are formed on the granules, then rupturing the incrustations by passing the granules between rolls set at a distance apart which is only slightly less than the size of the granules and without substantial reduction in the size of the granules, forming a bed of the coarsely crushed granules and passing an acid leach liquor through the bed, whereby high extraction rates and efficiencies are obtained without any packing of the bed.

Whiteware

Ceramic cast masses, 1914–1939. FOLKE SANDFORD. *Tek. Samfund. Göteborg. Avd. Kem. Fysik*, pp. 189–204; abstracted in *Chem. Zentr.*, **1940, II,** 2800.—S. reviews the manufacture and properties, the measurement of the viscosity, and physical and chemical principles of ceramic cast masses and the causes of the appearance of casting flaws. M.V.C.

Ceramic diaphragms permeable to water. Z. I. GLUSHANOK. *Keram. Sbornik*, No. 13, pp. 48–51 (1941).—The use of bentonite as bonding agent for mixes with a high grog content is recommended because porous bodies of high mechanical strength are obtained. The composition is special grog 80, bentonite 10, and clay 10%. The diaphragms should be cast in a gypsum mold. The slip should be liquefied with tannin (0.5%). M.V.C.

Ceramic insulating materials. H. THURNAUER. *Elec. Eng.*, 59 [11] 451–60 (1940).—T. reviews the preparation and the properties of electrical porcelain, high-frequency insulating materials, glass for electrical purposes, Mycalex, and vitreous enamels. 20 references. R.L.G.

Character of melting of feldspar. N. M. CHERVINSKII. *Keram. Sbornik*, No. 14, pp. 34–36 (1941).—The decrease in the specific gravity of porcelain in the interval between 1000° and 1100° is greater than the corresponding decrease in the specific gravity of feldspar. The process of vitrification, accompanied by a considerable lowering of specific gravity, ends at about 1200° for porcelain; the specific gravity of porcelain decreases little in the temperature interval from 1200° to 1300°. The process of vitrification of feldspar proceeds intensively to 1300°. In the interval between 1300° and 1400°, the specific gravity of feldspar remains unchanged while that of porcelain diminishes greatly. M.V.C.

Composition of high-voltage porcelain from Ural raw materials. I. YA. YURCHAK, I. M. TIKHOMIROV, AND Z. I. GLUSHANOK. *Keram. Sbornik*, No. 14, pp. 13–20 (1941).—Various attempts to manufacture high voltage porcelain ware from Ural raw materials are described, and the composition of some of the mixes prepared is given. M.V.C. 5 references, 6 figures.

Control of porcelain firing in tunnel kilns. G. A. KOVELMAN. *Keram. Sbornik*, No. 13, pp. 1–10 (1941).—To achieve satisfactory results in firing porcelain and insulators, a complete thermal control of the basic factors affecting the temperature conditions and gas atmosphere in the kiln is necessary. M.V.C.

Dependence of shrinkage and defects of porcelain ware on the position of mineral particles present in the ware. G. L. EFREMOV AND A. M. AKHVAN. *Keram. Sbornik*, No. 13, pp. 10–21 (1941).—The one-sided pressure that bodies undergo during technological working is dependent upon the character of the distribution of structural elements (mineral particles, pores, and water) in the bodies. The basic character of porcelain bodies is the fluidal structure (structure of flow) which is dependent upon the distribution of mineral particles in space. During working, the structural elements tend to lie perpendicular (of the long axis) to the mechanical pressure exerted upon them. Particles of kaolin have a scaly appearance. Sericite and hydrophilite found in large amounts in clays and kaolins have particles of the same character. Grains of quartz and grog and of fluxes (feldspar) have a broken appearance. The orientation of particles during casting is due to osmotic pressure occurring in the slip because of the absorption of water by the gypsum mold. The structural elements are distributed perpendicularly to the osmotic pressure, i.e., parallel to the walls of the gypsum mold. Bodies drawn through a press possess a homogeneous structure. Deformation and cracking of porcelain bodies depend on the degree of homogeneity of the body structure. Homogeneity, on the other hand, depends upon the method of shaping and the degree of working. The shrinkage of porcelain bodies depends on a series of factors, chief of which are as follows: (1) dehydration of kaolin, (2) degree of mullitization of the body, (3) degree of melting of easily fusible admixtures penetrating the body pores, and (4) structural characteristics (size, shape, and distribution of mineral particles, etc.) before firing. Porcelain bodies produce different shrinkages. This phenomenon depends on the distribution of structural elements, particularly kaolinite particles and pores formed because of the separation of water and air in the body. M.V.C.

Dimensions of fired porcelain products. N. M. CHERVINSKII. *Keram. Sbornik*, No. 15, pp. 17–19 (1941).—Observations on the shrinkage of a porcelain body in dependence on temperature and duration of firing are briefly analyzed. M.V.C.

Firing characteristics of insulators from Ural and Ukrainian raw materials. G. A. KOVELMAN. *Keram. Sbornik*, No. 15, pp. 9–16 (1941).—Preliminary tests showed that insulator bodies made from the Ural raw materials tend to swell less than those produced from Ukrainian raw materials and are thus less difficult to fire. To obtain high-grade porcelain, the bodies must be fired during vitrification in a reducing atmosphere. M.V.C.

Nepheline-syenite in hotel chinaware bodies. C. J. KOENIG. *Jour. Amer. Ceram. Soc.*, 25 [3] 90–93 (1942).—5 references, 6 figures.

Progress in the field of ceramic insulating materials for electrotechnical purposes. W. RATH. *Keram. Rundschau*, 48 [45] 371–72; [47] 394–96; [48] 403–404 (1940).—R. surveys the development during the last 10 years and the present status of ceramic insulating materials for all types of insulators, supports for coils, transformer windings, etc., bushings, and tubing. As in the case of metallic materials, the surface quality is of great importance with regard to the physical and mechanical properties, and fine cracks or other surface defects of porcelain or glass bodies must be avoided as the surface quality determines their mechanical strength. The glaze is of utmost importance as it increases the compressive strength and the resistance to sudden temperature changes of porcelain. The dielectric behavior, breakdown voltage, and electrical conductivity are discussed as dependent on shape, and tests made to elucidate distribution of the electric field, ion migration, and dielectric losses are described. Disturb-

ances in radio transmission can be caused by insulators of high electric field strength (pin insulators). 20 references. M.H.

Properties of ware vs. thermal history: I, Tests on vitreous china bodies and ware. J. H. KOENIG. *Jour. Amer. Ceram. Soc.*, 25 [2] 33–40 (1942).—17 references, 8 figures. **II, Tests on semivitreous china bodies and ware.** *Ibid.*, pp. 41–48.—10 figures.

Structure and properties of high-voltage porcelain. N. M. CHERVINSKII. *Keram. Sbornik*, No. 13, pp. 21–28 (1941).—The optimum temperature interval for firing porcelain insulators is from 1300° to 1320°C. At these temperatures, the temporary resistance to pressure reaches 322 kgm. per sq. cm. The resistance to pressure drops to 253 kgm. per sq. cm. at 1380°. The optimum duration of firing at the finishing temperature (1300°) is 5 hr. The shape and size of the quartz crystals and the degree of melting and crystallization are of importance in addition to the amount of glass and mullite present in the porcelain body. The porosity is also important as it influences the electrical stability of the body. C. concludes that, for increased mechanical properties, the porcelain should contain large amounts of argillaceous materials and less feldspar and should be fired at higher temperatures. Increase of the duration of the finishing firing is of little avail as the basic changes in the body take place during the first 5 or 7 hr. of firing (at 1300°). M.V.C.

PATENTS

Apparatus for manufacturing pottery. W. J. MILLER. U. S. 2,270,076, Jan. 13, 1942 (Oct. 4, 1939).

Decorated ceramic ware and method of making. W. E. PALMER (Taylor, Smith & Taylor Co.). U. S. 2,270,081, Jan. 13, 1942 (Feb. 16, 1939).—As a new article of commerce, ceramic ware which comprises a fired ceramic body, an opaque colored glaze on the ceramic body, and a differently colored pyrochemical reaction product under and combined with portions of such glaze to form for such ware a design of the underglaze type.

Method and apparatus for manufacturing pottery ware. W. J. MILLER. U. S. 2,268,717, Jan. 6, 1942 (Sept. 21, 1937). U. S. 2,270,075, Jan. 13, 1942 (Nov. 12, 1938).

Spark plug. F. J. BOLL (Globe-Union, Inc.). U. S. 2,269,076, Jan. 6, 1942 (May 20, 1940). T. G. MCDOUGAL (General Motors Corp.). U. S. 2,267,571, Dec. 23, 1941 (July 27, 1940). G. M. PAULSON. U. S. 2,269,682, Jan. 13, 1942 (June 15, 1940). U. S. 2,269,683, Jan. 13, 1942 (Nov. 23, 1940).

Spark plug. OTTO C. ROHDE (Champion Spark Plug Co.). Can. 401,147, Dec. 2, 1941 (May 18, 1939); in U. S. Feb. 13, 1936). G.M.H.

Spark plug. C. B. SAWYER (Brush Beryllium Co.). U. S. 2,268,903, Jan. 6, 1942 (April 30, 1940).—A spark-plug insulator composed of high-purity beryllium oxide having an apparent bulk density not in excess of about 0.35, the insulator being characterized by high thermal conductivity and a softening point above about 2000°C.

Talc-containing ceramic compositions. D. HAGAR. Brit. 541,695, Dec. 17, 1941 (Aug. 10, 1939).

Water closet. J. R. EVANS (Ne-Flex Mfg. Co.). U. S. 2,268,377, Dec. 30, 1941 (Dec. 31, 1940).

Equipment and Apparatus

Brewsterian angle and refractive indices. A. H. PFUND. *Jour. Optical Soc. Amer.*, 31, 679–82 (1941).—A procedure based on Brewsterian angle measurements yields refractive indices with uncertainties in the third decimal place. Errors due to surface films may be eliminated by making measurements only on freshly ground and polished surfaces. The procedure is applicable to black materials and has no upper limit. A.P.

Colorimetry and its use in the evaluation of the whiteness of porcelain and faience products. M. V. IVANOV. *Keram. Sbornik*, No. 14, pp. 36–42 (1941).—The theoretical background of colorimetric measurements is given, and their application in the measuring of the color of ceramic ware is discussed. M.V.C.

Continuous-reading electronic voltmeter for use with glass and other high-resistance electrode systems. C. J. PENTHER, F. B. ROLFSON, AND LOUIS LYKKEN. *Ind. Eng. Chem., Anal. Ed.*, 13 [11] 831–34 (1941).—A full-range, sensitive, low-current drain, continuous-reading electronic voltmeter is described. The meter has a range of ±2.11 volts and a sensitivity of ±0.001 volt over the entire range. It has been designed for the accurate determination of potentials of electrode systems having a resistance of 5000 megohms or less. Illustrated. F.G.H.

Determination of cloth area for industrial air filters. C. E. WILLIAMS, T. HATCH, AND L. GREENBURG. *Heating, Piping & Air Conditioning*, 12 [4] 259–63 (1940).—The area of cloth required is determined by the rate of air flow (cu. ft. per min.) and the rate of filtration required. The filtering velocity usually falls between 1.5 and 5.0 ft. per min., 3.0 being the most common value. The maximum filter resistance is usually held to 3 in. water gauge. The particle size of the dust, the porosity of the deposit on the filter, and the loading of dust are equally important factors. A resistance equation is given to correlate these factors. S.S.

Determination of the porosity of filters. H. WITZMANN. *Chem. Fabrik*, 12 [29–30] 345–53 (1939).—The porosity of filters is measured by different methods using gases and liquids. Methods of calculation are given, and the results for filters made from given materials using the different media are compared. The grain size of filters used varied between 260 and 17 μ, and the size of the corresponding pores varied between 100 and 13 μ. See "Elementary . . .", *Ceram. Abs.*, 21 [1] 28 (1942). J.M.N.

Electrically operated clay gun. A. F. MORGAN. *Iron & Steel*, 14 [1] 12–13 (1940).—The gun is suspended from a boom mounted on a column; it is adjusted to the plugging position by a hand-operated wheel, and remote control enhances safety. The tapping hole of a blast furnace can be plugged against the flow of iron without reducing the pressure of the blast. The entire plugging operation requires only 15 sec.; as no time is allowed for the clay to harden, a greater amount can be introduced. A normal charge is 6 cu. ft., but the gun has a capacity of 10 cu. ft. A scale graduated in cubic feet indicates the amount used. E.H.McC.

Electrostatic electron microscope and some applications to colloid chemistry. H. MAHL. *Kolloid-Z.*, 91 [2] 105–17 (1940).—The electrostatic electron microscope developed by M. in the Research Department of the Allgemeine Elektrizitäts-Gesellschaft is described. The apparatus is marked by great simplicity of construction and operation. On account of the electrostatic method of operation, the instrument is very little affected by variations in voltage, as shown by tests with pulsating current. The limit of resolution of the apparatus lies below 8 to 10 mμ. The use of a double projection lens makes it possible, even though the focal length is kept constant, to employ three degrees of enlargement on the same screen, of which the smallest enlargement serves for a survey. Special construction of the object holder permits the tipping of the object through a definite angle, so that stereoscopic pictures may be taken. Experimental examples of the possibilities of the application of the instrument to colloid chemistry are given. W.H.H.

Flashing calorimeter. A. A. MARKSON AND Y. A. OLSON. *Combustion*, 12 [11] 51–55 (1941).—The flashing calorimeter is a new instrument for indicating and recording the composition of very wet steam or other very wet vapor-liquid mixtures. It was developed to meet the specific need for a simple device capable of giving information on the composition of steam and water mixtures existing in boiler circuits under various conditions of operation. As such, it has already proved its value in several

investigations of circulation in connection with top tube trouble in sectional-header boilers. It is capable of furnishing data on the variation of quality of the mixture in boiler tubes, which is of prime importance in circulation studies. H.E.S.

Fluid drive for mining machinery. JOHN D. ROSEBROUGH. *Mining Congress Jour.*, **27** [12] 30–34 (1941).—Fluid drive is quite common on power transmissions of 5 to 3000 h.p. It provides a stepless variable-speed drive when used with a general purpose a.-c. motor. There are two types, a traction type as used on automotive equipment and a variable speed fluid coupling. The traction type simplifies starting equipment; the attached motor always starts unloaded. Overloads stall the drive but not the motor and jammed machinery is not violently broken. It can operate in dust-laden atmospheres. The variable-speed device gives a rapid control of acceleration and stepless control of speed from a constant-speed a.-c. motor. Machines using these types of drives are conveyers, loaders, hoists, haulage trucks, crushers, power shovels, draglines, fans, rod mills, aerial ropeways, etc. Fluid drive balances the load on motors on conveyers. Trucks on the Mesabi Range weighing 62,000 lb. have this drive; they start in the same gear that they are to run in. There is a total of 2,000,000 h.p. being transmitted by fluid drive in industry. W.D.F.

Furnace-temperature measurements and their interpretation. H. KREISINGER, B. J. CROSS, AND E. H. KENNEDY. *Combustion*, **12** [9] 37–42 (1941).—The exposed and the shielded aspirating types of thermocouples, as applied to the measurement of gas temperatures in pulverized-coal furnaces, are compared, and the advantages and limitations of each are discussed. While the latter permits more readings to be obtained without cleaning, it is concluded that, with proper care, there is little difference in the results for the two types. The calibration and the testing of couples for contamination are outlined, and the construction of a water-cooled mounting is described. Instructions for the use of both the shielded and the exposed couple are included. H.E.S.

Grinding ceramic materials in ball mills in a vacuum. S. E. VIRO. *Keram. Sbornik*, No. 15, pp. 34–40 (1941).—A vacuum has practically no effect on the grinding efficiency of ball mills using either the dry or the wet method. M.V.C.

High-speed photography and its application to industrial problems. E. D. EYLES. *Jour. Sci. Instruments*, **18** [9] 175–84 (1941).—E. considers the history, apparatus, and technique of high-speed photography and discusses in detail moderate-, intermediate-, and high-speed recordings and their applications. 84 references, 9 figures. J.L.G.

Infrared spectrometer for industrial use. W. H. AVERY. *Jour. Optical Soc. Amer.*, **31**, 633–38 (1941).—An infrared spectrometer has been designed for the routine identification of chemical compounds in industrial use where speed, convenience, and ruggedness, as well as accuracy, are of importance. A.P.

Interpretation of sieve tests. L. F. TAYLOR. *Ind. Chemist*, **16** [189] 285–87, 297 (1940).—A "wet-split" method of screen analysis is described and illustrated by logarithmic graphs. Its value in interpreting crushing and grinding problems is pointed out, and the most efficient method of grinding, such as roll crushing, gyratory and jaw crushing, and rod-mill or ball-mill grinding, can be selected. H.E.S.

Leeds & Northrup temperature indicator. ANON. *Machinery* [N. Y.], **47** [6] 170 (1941).—An improved portable instrument for thermocouple temperature measurements is shown. R.H.B.

Measurements of the breaking strength of lyogels. C. BAILEY. *Jour. Phys. Chem.*, **45**, 493 (1941).—Measurements are given of the maximum torque required to produce the accelerated rotation of a set of glass prongs dipping into bentonite gels of various concentrations. The apparatus has been described ("New . . .", *Ceram. Abs.*, **20** [7] 175 (1941)). Equations are given showing the relationship between breaking torque and clay content and between reaking torque and percentage of added electrolyte (K_2SO_4). R.A.G.

Mold construction. E. LONGDEN. *Iron & Steel*, **14** [11] 356–58; [12] 383–85; [13] 411–13; [14] 433–34 (1941).—L. discusses patterns and mechanical details. E.H.McC.

New data on drying of solids in air. O. KRISCHER. *Chem. Fabrik*, **12** [1–2] 23–25 (1939).—The drying of solids can be calculated when the capillarity of the material is not excessive and when the grain size, diffusion resistance, and capillarity of piled solids in addition to the usual data on the air conditions (temperature, humidity, and velocity) are known. Equations are given. See "Physical. . .," *Ceram. Abs.*, **18** [5] 134 (1939). J.M.N.

New scale-reading microscope. ANON. *Jour. Sci. Instruments*, **18** [9] 192–94 (1941).—The optical systems employed in the scale-reading microscope manufactured by Taylor, Taylor and Hobson, Ltd., are described in detail. 4 figures. J.L.G.

Physical photometer. R. P. TEELE. *Jour. Optical Soc. Amer.*, **31**, 696–704 (1941).—Photometry has been carried on by visual observations because there has been no physical apparatus which would respond to light as accurately as the human eye. A physical photometer which "sees" light in accordance with the I.C.I. luminosity factors has been constructed. Results are more accurate than those of visual observers in the measurement of light transmissions of colored filters. Luminous transmissions obtained with 22 spectrally selective glasses are compared with values computed from spectrophotometric data with I.C.I. luminosity factors. A.P.

Predicting dust concentration. FRANK B. ROWLEY AND RICHARD C. JORDAN. *Heating, Piping & Air Conditioning*, **12** [12] 699–702 (1940); **13** [2] 99–101 (1941).—The dust concentration levels in enclosed spaces are affected more by the rates at which (1) dust-laden air enters the space, (2) dust is produced in the space, (3) dust settles from the air, and (4) air is filtered than by the efficiencies of the filters themselves. Continuous and intermittent filtering processes are described. Typical examples of dust-concentration analyses are given. S.S.

Progress in the field of chemical equipment: I, Agitators; II, Mixing and kneading machines; V, Filters and presses. BRUNO WAESER. *Chem. Fabrik*, **12** [1–2] 25–30; [13–14] 153–57; [29–30] 353–57 (1939).—New theories and developments are reported, and recent patents in all countries are enumerated. See "Selection. . .," *Ceram. Abs.*, **21** [1] 20 (1942). J.M.N.

Protection of crushing machinery against foreign substances. RUDOLF SIEBERT. *Chem. Fabrik*, **12** [13–14] 157–60 (1939).—To prevent damage from foreign substances, such as pieces of iron, in ores or slags, rolls are protected by springs attached to a sliding frame, Symons crushers have a top frame which can be lifted against strong springs, and hammer mills have special grooves to catch steel pieces. The best protection is afforded by the removal of tramp iron by magnetic means. Several types of magnets are described. J.M.N.

Quartz photoelectric spectrophotometer. H. H. CARY AND A. O. BECKMAN. *Jour. Optical Soc. Amer.*, **31**, 682–89 (1941).—A spectrophotometer for measurements in the ultraviolet and visible regions, which incorporates a direct-reading potentiometer, electronic amplifier, rugged balance-indicating meter, and a quartz prism, is described. A.P.

Resistor furnace with some preliminary results up to 2000°C. R. F. GELLER. *Jour. Research Nat. Bur. Standards*, **27** [6] 555–66 (1941).—G. describes a laboratory furnace electrically heated with special oxide resistors. The atmosphere within the furnace is oxidizing, and definite heating and cooling schedules can be maintained. Results are given of preliminary tests in which ThO_2, ZrO_2, BeO, CeO_2, MgO, and Al_2O_3 were heated singly or in various combinations in the temperature range 1800° to 2000°C. R.A.H.

Role of induction heating in war production. FRANK T. CHESNUT. *Mech. Eng.*, **63** [12] 861–64 (1941).—Miscellaneous applications of induction melting furnaces are briefly reviewed. Illustrated. F.G.H.

Shuttle car haulage for metal mines. L. E. YOUNG. *Mining Congress Jour.*, 27 [11] 12–18 (1941).—Shuttle cars have been used by the coal industry since 1936 to transport the material from a continuous mechanical loader to main-line haulage. They run on rubber tires and use cables, trolleys, storage batteries, or Diesel engines. The cars have a bottom conveyer and thus do not need to be moved while loading. They have recently been installed in potash, limestone, salt, zinc and lead, and gypsum mines. To determine whether the cars can be used effectively the following factors must be considered: (1) hardness, etc., of the ore; (2) water on the floor or in the ore; and (3) dimensions of drifts, etc. In selecting equipment, factors to be considered are (*a*) capacity and dimensions of the car, (*b*) type and capacity of power units, (*c*) ease of steering, (*d*) brakes, wheels, etc., and (*e*) safety features. Factors for economic desirability include net load at least cost, maximum and most effective length of haul, and determination of whether waste, tools, supplies, etc., can be handled by this system. W.D.F.

Size grading applied to terminal velocity of fly ash and other dusts. H. H. BUBAR. *Combustion*, 12 [12] 36–37 (1941).—B. discusses the fallacy of attempting to determine the particle size of subscreen dust by ascertaining the terminal velocity by elutriation and urges instead that results be expressed in terminal velocity rather than particle size, a practice followed in Great Britain. H.E.S.

Slip mixers and agitators. ANON. *Ind. Chemist*, 16 [191] 345–46 (1940).—To impart a high degree of homogeneity to complex pottery bodies, various types of agitators and blungers are used at Josiah Wedgwood & Sons, Ltd. Latest equipment involves high-speed agitators that can be started up when the slip has settled thus making continuous use unnecessary. Neatness and cleanliness are also stressed with the new equipment. Installations were made by L. A. Mitchell, Ltd. Illustrated. H.E.S.

X-ray camera for materials of large grain size. C. WAINWRIGHT. *Jour. Sci. Instruments*, 18 [10] 203 (1941).—A translatory movement is added to the oscillation normally given to materials of large grain size. This promotes a more representative examination of the specimen surface. A simplified form of film holder is described. J.L.G.

SEPARATE PUBLICATION

Heat Transfer to Clouds of Falling Particles. H. F. JOHNSTONE, R. L. PIGFORD, AND J. H. CHAPIN. *Univ. Illinois Eng. Expt. Sta. Bull.*, No. 330, 55 pp. (1941).—Price 60¢.—An experimental and theoretical investigation was made for the purpose of obtaining information essential to the design of equipment for heating finely divided material falling through a gas. Such information may find wide application in the drying of powdery materials, such as sand, oxides, etc., in briquetting, and in carbonization of powdered coal and for spray driers. The theory of heat transfer by convection and radiation from the furnace wall to the gas through which the material falls and from the gas to the material is developed, and the heat-transmission coefficients are derived for different kinds of gases and materials. The results, which are then checked by experiments, confirm the fact that the rate of heat transmission from a hot furnace wall to clouds of falling particles is rapid. When the diameter of the furnace is small, the transmission from wall to gas and from gas to particles takes place principally by convection. The fraction transmitted by radiation increases rapidly with the size of the furnace and the temperature of the wall. The most important factor in the over-all transmission is the rate at which the particles are fed to the furnace. When radiation is small, the main effect produced by varying the feed rate is the changing of the ratio of particle area to wall area, thus changing the relative magnitudes of the thermal resistances at the wall and at the particles without altering appreciably the respective convection coefficients themselves. The coefficient of heat transfer by convection from gas to particles is a function of particle size and rate of fall and is nearly independent of temperature of the ambient gas. The coefficient of heat transfer by convection from wall to gas is apparently a function of the size of the furnace as shown by experiments. The radiation coefficient is a function of the concentration of particles in the cloud and of the shape of the furnace. The application of the theory is illustrated by an example of the design of a furnace used as a flash calciner for decomposing zinc sulfate hydrate into zinc oxide, water vapor, and sulfur dioxide. M.H.

PATENTS

Abrasion-resisting alloy. R. W. SCHLUMPF (Hughes Tool Co.). U. S. 2,268,427, Dec. 30, 1941 (Nov. 10, 1941).

Hard-metal alloy. R. W. SCHLUMPF (Hughes Tool Co.). U. S. 2,268,428, Dec. 30, 1941 (Nov. 10, 1941).

Jar mill. T. A. HIBBINS (Stevenson Co.). U. S. 2,267,697, Dec. 23, 1941 (Oct. 7, 1938).

Kilns, Furnaces, Fuels, and Combustion

Burning natural gas. R. H. GODEKE. *Power*, 85 [8] 560–62 (1941).—Illustrated. F.G.H.

Burning pulverized coal in rotary cement kilns. R. M. HARDGROVE. *Amer. Inst. Mining & Met. Engrs. Tech. Pub.*, No. 1390; *Mining Tech.*, 5 [6] 9 pp. (1941).—Pulverized coal is used in 85% of all cement kilns. Direct firing is replacing the bin system. A study was made of 41 wet and 47 dry plants; 60 were bin-fired, and 28 were direct-fired. The burning of more coal increases kiln output but decreases kiln efficiency. Firing rate plotted against output shows a closer grouping for wet kilns and some advantage for direct-firing in wet kilns. Most of the moisture should be dried out of the coals in the pulverizer. Good direct-firing practice has the following requisites: (1) primary air 2½ to 3 lb. per lb. of coal; (2) air temperature leaving pulverizer 160° to 220°F.; (3) velocity at burner tip 6000 to 8000 ft. per min.; (4) fineness of pulverized coal 80% passing 200-mesh; (5) extremely uniform delivery of coal to burner, obtained by automatic control of feeder and air temperature; and (6) very uniform control of secondary air, best obtained by automatic draft control. W.D.F.

Connecting draft gauges. W. H. PUGSLEY. *Power*, 85 [4] 268–69 (1941).—Illustrated. F.G.H.

Controlling characteristics of ash with special reference to Illinois and Indiana coals. J. HARRINGTON. *Combustion*, 12 [11] 41–45 (1941).—Investigations covering a large number of Illinois and Indiana coals have demonstrated that the acid-base ratio of the ash offers a reliable index to the fusion temperatures, and means were developed to control the ash characteristics by the addition of silica or lime to obtain the desired results. Approximately 2000 tons of coal per day are being treated with silica. The controlling influence in the production of objectionable clinkers is not the softening point of the ash but the point of fluidity or the spread between the softening and fluid temperatures. H.E.S.

Firing pottery by electricity. A. BERNARD HOLLOWOOD. *Electrician*, 127 [3314] 317–18 (1941).—The Wedgwood factory at Barlaston is the first pottery factory in Britain to depend entirely on electricity. Continental producers have pioneered the electrical ovens; Americans (aided by cheap supplies of natural gas) have developed gas firing. Electricity has been used for some years for firing enamel kilns. The hardening-on process, in which colors are fastened to the already glazed ware, requires a somewhat lower temperature than biscuit and glost firing. The extremely high temperature necessary in the firing of earthen-

ware and china (1100°C. for earthenware and 1200° to 1300°C. for china) proved too severe for ordinary heating elements. The success of the Wedgwood factory indicates that the early defects have been corrected. For temperatures of 1150° to 1200°C. the nickel-chrome alloy (Nichrome) and the iron-aluminum alloy (Kanthal) have been found to be suitable heating elements. For still higher temperatures, Globar or Silit resistors are employed. These are composite resistors containing silicon carbide and fire clay. Each oven has its particular arrangement of heating elements. Nichrome is usually set in coils or strips in the channels of the refractory walls. Globar or Silit resistors take the form of rods which extend across the interior surface. Electrical firing effects a considerable saving in that the use of saggers or earthenware containers (to protect the pottery from direct flame) is avoided. In the Barlaston factory both biscuit and glost firing are performed in one unit, a two-way kiln 273 ft. in length. The heating zone registers a maximum temperature of 1200°C. The tunnel is traversed by a subway to facilitate the correction of car troubles, and a special roofing enables elements to be exchanged. If, as has been proved possible in the U. S., it should become practicable to reduce firing time to about one day, kilns could be reduced in size and cost to the advantage of the small potter. A Rotoelec electrically heated kiln 22 ft. in mean diameter, with an output of 4000 dozen per week and having a consumption of 50 to 52 kw.-hr., and a special straight electric kiln 50 ft. long, with an output of 1100 dozen and having a consumption of 29 to 30 kw.-hr., are illustrated. H.K.R.

Gas flow in fuel beds. J. G. Bennett and R. L. Brown. *Jour. Inst. Fuel*, **13** [72] 232–46 (1940).—Combustion in fuel beds is a complex process which has defied attempts at exact quantitative expression. An aerodynamic process of study is described which involved construction of full-scale models. The fuel was replaced by lumps of starch-coated glass of similar size, and air impregnated with iodine vapor took the place of the gases. This afforded a means of studying the distribution of gas flow in a stationary bed. It was concluded that the distribution of flow through the bed is entirely controlled by the distribution of the packing. The importance of closely graded fuel in securing uniform flow has led to an increasing application of such fuels. Important developments may be expected in the use of less closely graded fuels in which controlled segregation is made to furnish the desired aerodynamic conditions. H.E.S.

Light-hydrocarbon analysis. J. J. Savelli, W. D. Seyfried, and B. M. Filbert. *Ind. Eng. Chem., Anal. Ed.*, **13** [12] 868–79 (1941).—Various methods for the analysis of mixtures of light hydrocarbons from methane through isopentane were studied and compared with regard to their accuracy and suitability for routine operations. The methods studied include the automatically controlled Podbielniak (Model L) column, the Ward method (utilizing low-pressure fractional distillation and condensation), a dew-pressure method for *n*-butane-isobutane mixtures, the determination of olefins by absorption in various reagents and by catalytic hydrogenation, and the determination of isobutene by absorption in various reagents and by its reaction with anhydrous hydrogen chloride. Synthetically prepared mixtures were used for the fundamental investigations. Various methods of sampling and handling liquefied light-hydrocarbon mixtures were studied and compared. Illustrated. F.G.H.

Natural gas analysis chart. C. T. Justice. *Power's Data Sheet*, No. 103; *Power*, **85** [2] 135 (1941).—Using Power's Data Sheet No. 103, the approximate composition of natural gas on a weight basis can be found if the heating value and specific gravity are known. The gas from which this chart was computed is a composite of many fields ranging in heating value from 930 to 1150 B.t.u. and in specific gravity from 0.570 to 0.650. Specific gravity, referred to air, and B.t.u. per cu. ft. at 30 in. Hg at 60°F. are usually known in a routine way to most plants that burn large quantities of gas. Since all the hydrocarbons present are included in the chart, the analysis obtained is quite complete. F.G.H.

Selecting chimney size for natural draft. J. G. Mingle. *Power*, **85** [4] 283–85 (1941).—Factors affecting the selection of chimney sizes are described in detail. Illustrated. See "Performance . . .," *Ceram. Abs.*, **18** [8] 220 (1939). F.G.H.

Shortcuts in combustion. Philip Swain. *Power*, **85** [7] 499–501 (1941).—S. explains the meaning of a flue-gas analysis and presents an easy way to figure theoretical air, actual air, and actual weight of flue gas per lb. of any fuel. Illustrated. F.G.H.

Significance of iron in coal used for combustion. A. H. Moody. *Combustion*, **12** [8] 22–26 (1941).—The significance of the presence of iron in mineral matter in coal used for combustion in the modern boiler is discussed. Mineral forms, iron and sulfur relationships, and pyrite distribution in the original coal are described. The changes in the chemical and physical properties of the ash during the course of combustion of coal are detailed. Limitations are imposed by the nature of ash upon the design and operation of large stoker- and pulverized-coal-fired boilers, and the degree of oxidation of the iron oxide in the ash at any given point in the furnace is a vital factor. Boiler-tube slagging and erosion of refractories depend upon the interrelated properties of the coal-ash slag: fusion temperature, flow temperature, viscosity, and iron content, especially ferric percentage. H.E.S.

SEPARATE PUBLICATIONS

Carbonizing Properties and Petrographic Composition of Upper Freeport Coal from Morgantown District, Monongalia County, W. Va., and of Lower Freeport Coal from Eastern Indiana County Near Cambria County, Pa. A. C. Fieldner, J. D. Davis, W. A. Selvig, D. A. Reynolds, R. E. Brewer, G. C. Sprunk, and C. R. Holmes. *U. S. Bur. Mines Tech. Paper*, No. 621, 77 pp. Supt. of Documents, Govt. Printing Office, Washington, D. C. Price 15¢. R.A.H.

Coal Paleobotany. Reinhardt Thiessen and George C. Sprunk. *U. S. Bur. Mines Tech. Paper*, No. 631, 56 pp. Supt. of Documents, Govt. Printing Office, Washington, D. C. Price 15¢.—The high lights of previous research on paleobotany of coal are presented. Coal contains an enormous amount of material in a remarkably good state of preservation, and it comprises constituents or components that invariably have retained some of the original plant structure. Many of these structures may be assigned to certain plants, and the identity of these plants and of the remains is discussed. The observations recorded are the outcome of work related to coal petrography, and no intensive work on paleobotany has been attempted. R.A.H.

Composition of Furnace Atmospheres Resulting from Partial Combustion of Gaseous Fuels. American Gas Assn. Committee on Industrial Gas Research. *Amer. Gas Assn. Bull.*, No. 11, 96 pp. (Aug., 1940). Price $1.25.—Changes in the amount of combustion air had the most effect on flue-gas composition. The total amounts of carbon and hydrogen in fuel gas are also important. Other variables have a minor effect. As air supplied decreased, CO and H_2 increased and CO_2 and H_2O decreased. The minimum amount of air required to support combustion ranged from 52 to 77% of the theoretical amount, depending on type of burner, furnace, and gas being burned. Flue gases contained practically no ethane, methane up to 1%, and illuminants 0 to 0.5%, the concentrations increasing with decrease of combustion air. All flue gases contained oxygen in concentrations up to 0.2%. The volume of flue products decreased almost in direct proportion to aeration decreases. The amount of each principal flue-gas constituent was found to be a function of the total amounts of carbon and hydrogen in fuel gas. CO and H_2 in flue gas remained in nearly constant ratio for any one fuel gas. At any given aeration, the ratio of CO to CO_2 in flue gases was practically constant for different gases. CO/CO_2 increased as combustion air decreased. Flue temperatures decreased from 3200° to 2700°F. as aeration was decreased from 100 to 70% for natural gas and 93 to 58% for manufactured gas. Heat

content in flue products and latent heat of water vapor decreased as aeration decreased, but potential heat of unburned combustibles increased rapidly; the net result was an increase of total heat in flue products in direct proportion to the decrease in aeration. A major part of the potential heat was due to H_2 and CO. The total heat in flue products at a given temperature and aeration, in per cent of heat in fuel, varied for different gases. Heat release per cu. ft. of combustion space decreased as aeration decreased, owing to the lengthening of the flames. Natural gas flames were longer than those of manufactured gas. S.S.

Heat Transfer by Radiation. W. J. WOHLENBERG. Engineering Experiment Station, Purdue University, Lafayette, Ind. Price 25¢. Reviewed in *Power*, **85** [3] 217 (1941).—A series of lectures presented at Purdue University, April, 1940, is reprinted. Topics covered are as follows: Nature of Radiation and Its Net Interchange between Surfaces of Solids, Spectral Distribution of Energy and Radiation from Gases, Equations Representing the Net Interchanges by Radiation Occurring in Systems Involving Gases With and Without Particles in Suspension, and Distribution of Energy in the Pulverized Coal Furnace Cavity. F.G.H.

PATENT

Electrically heated furnace. F. W. BROOKE (Swindell-Dressler Corp.). U. S. 2,262,691, Jan. 6, 1942 (March 20, 1939).—In a furnace, the combination with the furnace chamber of electrical heating means therefor comprising a hollow porous resistor of refractory material within the chamber and means for closing cracks and joints in the hollow resistor comprising means for passing into the hollow resistor a gas including a carbon constituent which cracks at high temperatures and deposits solid carbon thereby formed in the cracks and joints.

Geology

American bauxite supplies. ANON. *Economist*, **141** [5127] 615–21 (1941).—The U. S. is one of the most important bauxite-producing countries. In 1940, 445,981 metric tons were produced in Arkansas, Alabama, and Georgia, and an increase of 50% is anticipated. The chief source of imported bauxite is Surinam, which supplied 585,993 tons or 93% of all imports, much smaller quantities being supplied by British Guiana and the Dutch East Indies. Two new plants at Paranam were opened in February, 1941, producing 150 tons of dry bauxite per hr. British Guiana is expected to produce 1,000,000 tons a year before long, but the bulk has hitherto gone to Great Britain and Canada. The huge bauxite deposits at Poços de Caldes in Brazil are of good quality but cannot be shipped as the freightage alone is more than twice the price of domestic bauxite in the U. S. A.B.S.

Base exchange of the clay mineral montmorillonite for organic cations and its dependence upon adsorption due to van der Waals' forces. STERLING B. HENDRICKS. *Jour. Phys. Chem.*, **45**, 65–81 (1941).—The structures of several organic molecules were studied. R.A.G.

Bauxite. LAWRENCE LITCHFIELD, JR. *Chem. Industries*, **48** [2] 154–59 (1941).—The chemical and physical properties of bauxite are discussed. Pure aluminum oxide (corundum) is used as an abrasive in the form of emery but has been largely superseded by artificial abrasives from fused bauxite. For this purpose, bauxite high in alumina and low in impurities is required. When bauxite is fused with lime, a cement is produced which is relatively resistant to attack by acids and chemicals, which will withstand higher temperatures than Portland cement, and which attains its maximum hardness in about 24 hr. E.D.M.

Bauxite, vital strategic ore accessible to all powers. ANON. *Science News Letter*, **40** [24] 380 (1941).—Bauxite deposits occur in North and South America, India, Africa, Australia, Netherlands Indies, U. S. S. R., Hungary, Italy, Yugoslavia, France, Greece, Rumania, and Dutch Guiana. G.A.K.

Bradleyite, a new mineral, sodium phosphate-magnesium carbonate. JOSEPH J. FAHEY AND GEORGE TUNELL. *Amer. Mineralogist*, **26**, 646–50 (1941).—An anisotropic fine-grained mineral (bradleyite), associated with shortite and carbonaceous clay, was found in the drill core of the John Hay, Jr., Well No. 1 in Sweetwater Co., Wyo., at a depth of 1342 ft. 10 in. Chemical analysis shows that the formula is $Na_3PO_4 \cdot MgCO_3$. X-ray powder diffraction photographs of bradleyite show that it does not contain $MgCO_3$ in the form of magnesite. The spacings and intensities of the lines of the X-ray powder photograph of bradleyite are tabulated to aid in its future identification. W.D.F.

Chemical treatment of bentonitic suspensions and the relation to the heaving shale problem. H. H. POWER AND CHARLES R. HOUSSIERE, JR. *Amer. Inst. Mining & Met. Engrs. Tech. Pub.*, No. 1401; *Petroleum Tech.*, **4** [6] 16 pp. (1941).—An investigation of the effect of chemicals on Wyoming bentonite preceded a study of heaving or caving shale which may prevent oil-well drilling. The particles were fractionated on a supercentrifuge. The viscosity of the dilute suspensions used was measured on a Stormer viscosimeter as this was the best means for comparing resistance to shear under various conditions. Filtration on Baroid filtrate testers was used to determine the rate of loss of water due to permeability of the bentonite cake. KCl, NaCl, HCl, Na_2SO_4, and NaOH were added to the suspensions in concentrations of 2 *N*, 1 *N*, and 0.5 *N*. Conclusions are as follows: (1) effective positive ion adsorption is in the order Na, K, H; (2) NaOH enmeshes water, while NaCl does not; (3) with both 1 *N* KCl and 1 *N* HCl, viscosity and filtration are different than when each is used separately; (4) HCl has more effect than KCl; (5) with constant filtration rates, viscosity changes may indicate ionic structures; (6) a relation exists between peptization and particle orientation; (7) in the two systems, 1 *N* NaCl–1 *N* Na_2SO_4 and 2 *N* NaCl–2 *N* Na_2SO_4, viscosity and filtrate relationships are reversed; (8) with salts, the reduction of viscosity is due to the number and kind of ions; (9) viscosity varies in inverse proportion to particle size; (10) "bridging" of particles is important in filtration; (11) smaller particles retard pH change; and (12) these conclusions are not tenable for higher reagent concentrations. W.D.F.

Colloidal clays: II. E. A. HAUSER AND D. S. LE BEAU. *Jour. Phys. Chem.*, **45**, 54–65 (1941).—The cataphoretic velocity of colloidal bentonite fractions increases with decreasing particle size; with constant particle size, it decreases with increasing concentration. The viscosity in extreme dilutions is independent of particle size but increases progressively with concentration and with particle size at higher concentrations. The apparent specific gravity of bentonite at low concentrations is lower than that of dry bentonite; at high concentrations it is greater. Explanations are given. For Part I see *Ceram. Abs.*, **19** [2] 52 (1940). R.A.G.

Crystal structure of α-AlOOH and α-FeOOH. W. HOPPE. *Z. Krist.*, **103** [2] 73–90 (1940).—The structure of diaspore as determined by Ewing (*Ceram. Abs.*, **14** [7] 171 (1935)) has been reinvestigated to measure the u-parameter more exactly as well as the intensities of the reflections (h00). Aikinite has also been studied. R.L.G.

Defense of bauxite. ANON. *Chem. Age* [London], **45** [1170] 275 (1941).—The U. S. Army has intervened in Surinam (Dutch Guiana) to safeguard the supply of bauxite which provides over 60% of the requirements of the U. S. aluminum industry and, together with the deposits in British Guiana, constitutes the most important source of this precious war material in the Western Hemisphere. The Netherlands Government and the Government of Brazil are cooperating in safety measures, and an

exchange of information on the subject has been arranged among the three powers concerned. A.B.S.

Degree of dispersion of clays: IV, Shapes of clay particles. C. E. MARSHALL. *Jour. Phys. Chem.*, **45**, 81–93 (1941).—Investigation of the dityndallism caused by the flow of various clay minerals is used to determine the shapes of the clay particles, which are shown to be in the form of plates. For Part III see *Ceram. Abs.*, **20** [1] 28 (1941). R.A.G.

Fuller's earth of Gaura in Transylvania. R. GRENGG. *Keram. Rundschau*, **48** [50] 421–24; [51] 434–36 (1940).—Naturally occurring clays which have such a high content of the finest swelling constituents that they feel greasy to the touch when moistened with water are called fuller's earth. They can absorb considerable amounts of fats and oils and thus show, to a certain extent, properties similar to those of true washing soaps (such as American bentonite). They occur in larger deposits in Algiers and Tunisia and in Transylvania in Europe. The geological and geographical characteristics of the deposit near the village of Gaura are described; dry earth swells to 15 times its volume when mixed with water. The physical distribution of grain sizes is given in the following table:

Designation (according to Correns)	Grain-size limits (mm.)	%
Gravel	2–5	0.55
Coarse sand	0.2–2	9.55
Fine sand	0.05–0.2	8.95
	0.02–0.05	0.23
Coarse clay	0.01–0.02	2.15
	0.007–0.01	2.15
	0.003–0.007	7.64
Fine clay	0.0013–0.003	6.44
	0.0008–0.0013	8.12
	0.0003–0.0008	22.76
Colloidal clay	Below 0.0003	31.45

The viscosity is very high due to the high content of colloidal constituents; a suspension with 5% is thickly viscous, and one with 7% is pasty. A high-percentage suspension shows birefringence under the microscope. A purified fuller's earth (freed of coarse particles) has the following composition in the water-free state: 74.33 SiO_2, 15.13 Al_2O_3, 5.07 Fe_2O_3, 31.70 MgO, and 1.75% CaO. The material is used mostly as washing and cleaning soap but also, mixed with soda, soap, crude oil, petroleum, etc., as a substitute for drilling oil; great care must be taken, however, to prevent rusting of machines and tools. M.H.

Magdalen Islands. F. J. ALCOCK. *Trans. Can. Inst. Mining & Met.*, **44**, 623–49 (1941).—The geology and mineral deposits of the Magdalen Islands are described. Residual pyrolusite is a possible source of manganese. Much impure gypsum occurs on the Islands. G.M.H.

Methods of evaluating difficultly fusible materials: IV, Base-exchange capacity of refractory clay. L. BELLADEN. *Metallurgia Ital.*, **32**, 236–38 (1940); abstracted in *Chem. Zentr.*, **1941, I**, 260.—Numerous tests were made on refractory clays of varying plasticity; they showed that the method of Vageler for determining the capacity for the exchange of bases is superior to the method of Graham and Sullivan (*Jour. Amer. Ceram. Soc.*, **21** [5] 176–83 (1938)) of dissolving with 1 *N* NH_4 acetate solution, not only because it is quicker but also because it gives better values for the plasticity and is more dependable. For Part II see *Ceram. Abs.*, **15** [11] 346 (1936); see "Methods . . ." *ibid.*, **17** [4] 143 (1938). M.V.C.

Properties of soluble silicates: II, Uses. C. H. JEGLUM. *Chem. Industries*, **49** [6] 604–10 (1941).—J. presents a detailed account of the uses of soluble silicates as adhesives, as cements (where other materials are combined with silicate to react chemically with the formation of a mass having strong bonding properties), as sizes and coatings, on porous metal castings to make them usable, in paints for stucco and concrete buildings, in detergents for the deflocculation of removed dirt, and in ceramics for the deflocculation of clay slips. 15 references. For Part I see *Ceram. Abs.*, **21** [1] 27 (1942). E.D.M.

Pyrope garnet vs. ruby spinel in Kansas. BENEDICT P. BAGROWSKI. *Amer. Mineralogist*, **26**, 675–76 (1941).—Serpentinized peridotite near Stockdale, Riley Co., contains a mineral which has been called ruby spinel. This mineral has a chemical analysis very close to that of pyrope garnet. W.D.F.

Use of German talc in ceramic masses. ANON. *Keram. Rundschau*, **48** [52] 445–46 (1940).—An attempt was made to determine whether German talc containing a comparatively high percentage of Ca and Fe could be used successfully in refractories, acidproof pottery, and masses for porous insulating bodies. The chemical composition of the talc is 56.5 SiO_2, 27.4 MgO, 5.8 Al_2O_3, 2.8 CaO, 2.1 Fe_2O_3, and 5.4% loss on firing. Mixtures of 80 to 30% kaolin with 20 to 70% talc showed that 30 to 40% talc can be used; a lower shrinkage and a slightly higher porosity were obtained with the German talc. More than 40% can be used only in masses with low firing temperatures, as the presence of Ca and Fe results in strong deformation. A porous insulating body was made with 40 refractory fat clay, 40 German talc, and 20% finely ground alumina fired at Seger cone 13. It fires yellow to brown, has a shrinkage of 8%, and has a porosity of 29%. For a pottery mass with a possible basic composition of 48 SiO_2, 37 Al_2O_3, and 15% MgO, 60% talc was used. This mass was yellow. It is used for acid containers. For refractory masses, the best batch was found to be 20 refractory fat clay, 25 lean clay, 14 German talc, and 41% fire clay; the latter was added in sizes ranging from dust to grains 3 mm. in diameter. The mass had a porosity of 15% and a shrinkage of 5%. Rods 26 x 2 x 1 cm. showed, on loading, a deflection of 10 mm. A sintered magnesite was made of 82 MgO, 10 SiO_2, 4 Fe_2O_3, 2 Al_2O_3, and 2% CaO, to which 20% German talc was added. Attention is called to the differences in the composition of German talc, and the selection must be made carefully according to the given case. M.H.

Use of the raw materials of Middle Asia in porcelain and faience production. L. A. VIKHIREVA, *Keram. Sbornik*, No. 13, pp. 29–37 (1941).—The large deposits of agalmatolite of the kaolinite and pyrophyllite varieties in the Ak-Tash region are discussed, and the testing of these materials for porcelain and faience production is described. M.V.C.

Volume of sediment of kaolin powders dispersed in electrolyte solutions: II. G. G. KANDILAROV. *Kolloid-Z.*, **91** [1] 56–61 (1940).—K. compared the variations in volume of sediment of kaolin in solutions of sodium salts, such as NaCl, Na_2SO_4, Na_2HPO_4, and Na_3PO_4, which have one, two, or three anions. The concentration-sediment volume curves ordinarily show slight maxima near the beginning. With further increase in concentration of the univalent and bivalent Cl and SO_4 anions, the volume of sediment of kaolin strongly decreases to a broad minimum, after which a rise is observed. The sodium sulfate *C-SV* curve is somewhat displaced downward and toward the left. In the corresponding weak reacting Na_2HPO_4 solutions, the decrease in volume of sediment is much less. In strongly alkali Na_3PO_4 solutions the *C-SV* curve is displaced downward, and its right branch shows a very steep rise, which is related to the corresponding steep rise of the sodium hydroxide *C-SV* curve. The greatest changes in volume of kaolin sediment were observed for solutions of HCl and NaOH (with strongly acid or alkaline salts), e.g., the volume of kaolin sediment for concentrations of 10 to 500 millimols per liter of the acid salt solutions increased about 40%, while its structure was loosened, but the volume of kaolin sediment in NaOH solutions was almost doubled. These changes are to be ascribed mainly to the hydrogen or hydroxyl ions. The fact that the changes in the volume of kaolin sediment in strongly hydrolyzed $AlCl_3$ solutions are similar to those with low concentrations of HCl solutions, as well as the fact that the direction of the sodium hydroxide *C-SV* curve is the same as the right branch of the Na_3PO_4 solutions, shows that the observed volumes of sediment are additive values, at least at these concentrations. The volume of sediment with the acid $AlCl_3$ solutions is due to the simultaneous effect of the Al, Cl, and

H ions; in the case of the strongly alkali Na_3PO_4 solutions, it is due to the simultaneous action of the Na, HPO_4, and OH ions. The general course of variations in volume of kaolin sediment with the strongly alkaline Na_3PO_4 solutions is entirely different from the course with the acid $AlCl_3$ solutions. The cause for this phenomenon may be found in the opposed electrically charged trivalent Al, HPO_4, or PO_4 ions and the H or OH ions which are present on account of hydrolysis. For Part I see "Sedimentation. . .," *Ceram. Abs.*, 19 [9] 224 (1940). W.H.H.

SEPARATE PUBLICATIONS

Geology of the "Martic Overthrust" and the Glenarm Series in Pennsylvania and Maryland. ERNEST CLOOS AND ANNA HIETANEN. *Geol. Soc. Amer. Special Paper*, No. 35, 207 pp. (1941).—An attempt is made to solve a fundamental and controversial problem of Appalachian geology by various methods of attack in field and laboratory, including application of the modern techniques of petrofabric analysis, rock analyses, and residue analyses. Many petrographic, metamorphic, and structural data are discussed. The authors conclude that a large portion of the crystalline rocks (known as the Glenarm series and including the Wisshickson schist) that crop out in the Piedmont province of Pennsylvania and Maryland cannot be pre-Cambrian, as previously interpreted, but must be metamorphic rocks of early Paleozoic age. A.C.B.

Occurrence and Origin of the Titanium Deposits of Nelson and Amherst Counties, Va. C. S. Ross. *U. S. Geol. Survey Professional Paper*, No. 198, 59 pp. Supt. of Documents, Govt. Printing Office, Washington, D. C. Price 25¢.—The Roseland titanium district of Virginia has long been the world's principal source of rutile, and it also contains important deposits of ilmenite. R. describes the minerals and mineral relationships of the occurrences of both the rutile and ilmenite. These indicate that rutile, ilmenite, and apatite, together with a large group of accessory minerals, were introduced into an essentially pure feldspar rock (anorthosite) by hydrothermal or vapor-phase magmatic emanations. The physical chemistry of such an origin is considered, and a paragenetic history is suggested. The significant mineral relationships are illustrated by 19 plates. R.A.H.

Chemistry and Physics

Ceriometric method for the determination of sulfates. J. P. MEHLIG AND K. R. JOHNSON. *Chemist-Analyst*, 30 [4] 76–78 (1941).—In the ceriometric method for the determination of sulfates, oxalate equivalent to the sulfate is titrated with ceric sulfate. The method is less tedious than the gravimetric method but is subject to more interference and is best suited for routine control analyses. Compared with the permanganate titration method, the ceriometric method offers easier preparation and a more stable solution, simple titration with no side reactions, and noninterference by chlorides. A.P.

Dielectric strength of mixed crystals. H. FRÖHLICH. *Proc. Roy. Soc.* [London], **A178**, 493–98 (1941).—The increase in breakdown strength of ionic crystals due to the admixture of foreign atoms is calculated quantitatively. A.P.

Fluorescent tests for beryllium and thorium. CHARLES E. WHITE AND C. S. LOWE. *Ind. Eng. Chem., Anal. Ed.*, 13 [11] 809–10 (1941).—The reagent 1-amino-4-hydroxyanthraquinone serves well for detecting beryllium in alkaline solutions and thorium in acid solution. In the case of beryllium, the test is less sensitive but more specific than with morin and much more definite than with quinalizarin. In application to thorium the sensitivity is not so great as might be desired but is sufficient for many practical purposes and provides a vivid color reaction for the identification of this element. Several hydroxyanthraquinones were tested with these elements, and the only other one found to present analytical possibilities was 1,8-dihydroxyanthraquinone. Although all possibilities have not been tried, it seems obvious that tests with metallic ions may assist in identifying the location of groups in the anthraquinones. See *Ceram. Abs.*, 18 [9] 255 (1939). F.G.H.

Hydrofluosilicic acid method for determination of quartz. F. H. GOLDMAN. *Ind. Eng. Chem., Anal. Ed.*, 13 [11] 789–91 (1941).—A method for the analysis of the quartz content of granular material using hydrofluosilicic acid has been extended to include "settled dusts." Mechanical losses and losses due to the solution of quartz by the reagent have been greatly reduced. There is a substantial saving of time in an analysis which may take as long as 2 weeks to complete. Illustrated. F.G.H.

Manganometric determination of calcium. A. IEVIŅŠ AND F. OŠIS. *Z. Anal. Chem.*, 120 [11–12] 401–10 (1940).—The titration of calcium oxalate with $KMnO_4$ solution gives very good results if the oxalate is precipitated from dilute, salt-free solutions. With larger amounts of salts, a reprecipitation is necessary. The washing of the precipitate must be done with saturated calcium oxalate solution. R.L.G.

Naming spectroscopic analytical methods. M. G. MELLON. *Jour. Optical Soc. Amer.*, 31, 648–50 (1941).—Current literature on analytical applications of spectroscopy seems confused in the usage of terms. M. suggests a possible nomenclature. The naming of a technique should bring out the method of separating the desired constituent and the property measured. Examples are gravimetric electrodeposition, titrimetric precipitation, colorimetric volatilization, and absorptive spectrometric precipitation. In an account of a research, writers should leave no uncertainty about the kind of apparatus used and the conditions of use. A.P.

Photometry in spectrochemical analysis. W. C. PIERCE AND N. H. NACHTRIEB. *Ind. Eng. Chem., Anal. Ed.*, 13 [11] 774–81 (1941).—A study has been made of the photometric methods used in spectrochemical analyses, and the procedures which give optimum results are listed. Included in this study are the methods for plate calibration, the variations in plate gamma with wave length, the effect of background and methods for correcting, and the accuracy obtained in photometric measurements of intensity ratios. Working curves and the factors governing the selection of an internal standard are discussed. Graphical computation methods are described. Criteria for evaluation of a photometer are given, and the construction of a satisfactory instrument is described. Illustrated. F.G.H.

Separation of bismuth from lead with ammonium formate. SILVE KALLMANN. *Ind. Eng. Chem., Anal. Ed.*, 13 [12] 897–900 (1941).—The sodium formate method, proposed by Benkert and Smith for the quantitative separation of bismuth from lead, shows definite advantages over other procedures, but there are obstacles to its general adoption. A new procedure is proposed, suited for both the quantitative separation and determination of bismuth and lead and for the quantitative separation of small amounts of bismuth from large amounts of lead. The nitric acid solution of bismuth and lead is neutralized with ammonia and ammonium carbonate. Ammonium formate is added, and the precipitate of basic bismuth formate is filtered off, washed with hot water, reprecipitated, and finally ignited to the oxide. The precipitate can also be dissolved in hydrochloric acid, and bismuth can be determined as oxychloride. If the bismuth formate precipitate is very small, it can be dissolved in dilute sulfuric acid and can be determined colorimetrically with potassium iodide. Lead is precipitated in the filtrate of the bismuth formate as chromate with potassium or ammonium dichromate. F.G.H.

Separation and determination of lead with salicylaldoxime. W. B. LIGETT AND L. P. BIEFELD. *Ind. Eng. Chem., Anal. Ed.*, 13 [11] 813–15 (1941).—Lead can be determined by precipitation as lead salicylaldoximate, weighing the resulting precipitate after drying at 105°C.,

and calculating the amount of lead on the basis of the formula $PbC_7H_5O_3N$. From nitrate solutions, precipitation begins just above pH 4.8 and is complete at 6.9. The precipitate obtained at pH 6.9, however, does not have the composition represented by the formula $PbC_7H_5O_3N$. Precipitation must be carried out at pH 8.9 or above to obtain a compound to which the theoretical factor of 0.6053 for lead in lead salicylaldoximate can be applied. The effect of acetate concentrations as low as 0.05 molar is to increase the pH necessary for the beginning of precipitation, for complete precipitation, and for complete precipitation as lead salicylaldoximate. This increase is about 0.5 pH unit. Lead can be separated as the salicylaldoximate from silver, cadmium, and zinc in strongly ammoniacal solutions. Salicylaldoxime reagent cannot be used in conjunction with high ammonia concentrations to separate lead from one or more of the following metals in solution: copper, nickel, cobalt, bismuth, iron, magnesium, manganese, and mercury. Illustrated. F.G.H.

Silicic acid gels: XI, Effect of concentration of silica upon the time of set. CHARLES B. HURD AND HOWARD E. SHEFFER. *Jour. Phys. Chem.*, **45**, 588 (1941).—With gels made from sodium silicate and acetic acid at a pH range of 4.8 to 6.5, the time of set of these mixtures at a fixed temperature and constant silica concentration is proportional to the H-ion concentration for each concentration of silica. The time of set appears to be inversely proportional to the square of the silica concentration at fixed temperature and pH. With some assumptions, indications of a third-order reaction are obtained. The energy of activation is nearly independent of the concentration of silica, being about 16,500 calories. For Part X see *Ceram. Abs.*, **20** [8] 204 (1941). R.A.G.

Space filling in SiO_2 modifications. H. TERTSCH. *Z. Krist.*, **103** [2] 96–111 (1940).—The "space-filling forms" have been determined graphically for the various modifications of crystalline silica. Here, as in many other crystals, the densest packing of spheres is not attained. The SiO_2 molecule has a form of tetrahedral character with the Si ion at the center of gravity. The Si form is tetrahedral and remains unchanged in all modifications, whereas the O form is lenticular and varies with the modifications. The O form further shows different appearances in the low and high forms. These observations are discussed on the basis of the Bohr-Kossel structures of atoms and ions. R.L.G.

Spectrochemical analysis of alkali products. E. A. BOETTNER. *Ind. Eng. Chem., Anal. Ed.*, **13** [12] 861–64 (1941).—B. gives details for the determination of sodium hydroxide, soda ash, sodium bicarbonate, sodium chloride, calcium carbonate, calcium hydroxide, and calcium chloride. Illustrated. F.G.H.

Spectrochemical determination of lead, cadmium, and zinc in dusts, fumes, and ores. H. I. OSHRY, J. W. BALLARD, AND H. H. SCHRENK. *Jour. Optical Soc. Amer.*, **31**, 627–33 (1941).—A spectrochemical method for the determination of lead, cadmium, and zinc in samples of dusts and fumes collected with the Barnes electrostatic precipitator is described. A.P.

Spectrographic studies of coprecipitation. Fourth-period elements with barium sulfate and copper and zinc with lead sulfate. LOUIS WALDBAUER, F. W. ROLF, AND H. A. FREDIANI. *Ind. Eng. Chem., Anal. Ed.*, **13** [12] 888–89 (1941).—The coprecipitation of iron, cobalt, nickel, chromium, and manganese with barium sulfate and of copper and zinc with lead sulfate was studied spectrographically. All precipitates of barium sulfate were prepared by the method of Popov and Neumann, which consists of the addition of the sulfate ions to the solution of the barium ions. It was found that cobalt and nickel do not coprecipitate with barium sulfate, whereas iron, manganese, and chromium do. Copper and zinc coprecipitate with lead sulfate. F.G.H.

Strongest lines of singly ionized atoms. W. F. MEGGERS. *Jour. Optical Soc. Amer.*, **31**, 605–11 (1941).—Each chemical element is characterized by Z totally different atomic spectra, where Z is the atomic number. The first atomic spectrum (represented by I following the chemical symbol) is characteristic of neutral atoms, the second is ascribed to atoms that have lost one outer electron, etc. A table presenting the wave lengths and other data on the strongest lines in the second spectra of chemical elements may be compared with a similar listing for neutral atoms (see "Physical . . .," *Ceram. Abs.*, **20** [7] 183 (1941)). A.P.

Structure of tricalcium aluminate. HOWARD F. MCMURDIE. *Jour. Research Nat. Bur. Standards*, **27** [6] 499–505 (1941).—The work on the structure of $Ca_3Al_2O_6$ is reviewed. McM. shows that $Ca_3Al_2O_6$ has a cubic unit cell having $a_0 = 15.24$ a.u., with 24 mol. present. A comparison with other compounds with similar patterns shows that the metal atoms are arranged at or near the centers and corners of 3.81-a.u. cubes. No deductions can be drawn with regard to the oxygen arrangement from a consideration of the strong lines only. As there are many compounds which give patterns whose strong lines are those of a body-centered cube with 3.81-a.u. edges, it is suggested that the pattern obtained by Brownmiller (*Ceram. Abs.*, **18** [1] 8 (1939)) from slowly quenched glasses of Portland cement compositions was the result of submicroscopic quench growths of metastable crystalline compounds. R.A.H.

Water glass. BRUNO SCHWEIG. *Glass Ind.*, **22** [10] 436–39; [11] 476–79 (1941); see *Ceram. Abs.*, **21** [2] 51 (1942). A.P.

PATENTS

Manufacture of zinc oxide. C. R. BERINGER (C. R. Beringer, Eugen Horvát, Ernst Horvát, Ferdinand Solt, and Alfred Spiegel). U. S. 2,269,355, Jan. 6, 1942 (Sept. 26, 1936).

Producing improved titanium pigments. J. E. BOOGE (E. I. du Pont de Nemours & Co.). U. S. 2,269,139, Jan. 6, 1942 (April 27, 1938).

Production of zirconium and titanium oxides. W. O. H. SCHORNSTEIN. Brit. 541,343, Dec. 3, 1941 (May 21, 1940).

Zinc aluminate pigment and paint and method of making. L. E. BARTON. U. S. 2,269,508, Jan. 13, 1942 (March 16, 1939).—The method of making an essentially pure, opaque, white, pigmentary zinc aluminate which consists in making an intimate mixture of zinc oxide and gamma-aluminum oxide and then heating the mixture in the temperature range 750° to 1400°C. until the zinc aluminate is formed.

General

Aspects of underground ventilation. RAYMOND MANCHA. *Mining Congr. Jour.*, **27** [11] 27–30 (1941).—The chief requirement of mine ventilation is the maintenance of an adequate flow of air through the mine at the least possible cost. The following points should be considered: (1) Underground pressure surveys are desirable. (2) More accurate air-volume measurements should be made. (3) Air-velocity surveys are an easy method of checking conditions roughly. (4) There can be too many or too few airways. (5) Leaky stoppings should be fixed. (6) Regulators are put on the far end of a split. (7) A booster fan on a split is advantageous but needs careful location and regulation. (8) Air currents are best split close to the place where the air enters the mine and reunited where the air leaves the mine. W.D.F.

Boiler embrittlement protection. CYRUS W. RICE. *Combustion*, **12** [7] 37–39 (1941).—In reviewing the present knowledge with regard to embrittlement protection and prevailing practices, R. offers explanations for certain apparently conflicting experiences and shows how, under different circumstances, some salts may either accelerate or prevent embrittlement. H.E.S.

Compensation of occupational diseases. L. TELEKY. *Jour. Ind. Hyg. & Toxicol.*, **23**, 353–73 (Oct., 1941).—T.

reviews the subject of compensation laws for occupational diseases, including principles, history, types of laws, and a classification of the laws of the various states with reference to diseases and disabilities covered and details of administration. The diseases recognized are presented in tabular form for England, Germany, 13 states of the U. S., Puerto Rico, and the International Labor Office. F.S.M.

Defense of professional status of ceramists. GEORGE J. EASTER. *Bull. Amer. Ceram. Soc.*, **21** [1] 9–10 (1942).

Doctor in court. B. L. SHIESSTAG. *Bull. N. Y. Acad. Med.*, **17**, 692–96 (Sept., 1941). G. Z. MEDALIE. *Ibid.*, pp. 697–705. C. G. HEYD. *Ibid.*, pp. 706–14. F.S.M.

Dropping mercury electrode for lead analysis. E. C. BARNES AND H. W. SPEICHER. *Jour. Ind. Hyg. & Toxicol.*, **23**, 397–407 (Oct., 1941).—The operation of the dropping mercury electrode is described. Necessary precautions for the accurate determination of small quantities of materials and the use of the Fisher Electropode for the determination of lead are given. Good accuracy was attained in the lead analysis of fumes from soldering operations. The time required for such analyses is quite short compared with that required for other methods. F.S.M.

Education of engineers. C. E. INGLIS. *Engineer*, **172** [4478] 319–20; [4479] 327–28 (1941).—I. discusses the present educational methods in engineering in England, pointing out defects and suggesting changes under the following headings: (1) mathematics, (2) the humanities in relation to engineering education, (3) examinations, (4) workshop training, (5) drawing-office training, and (6) research and post-graduate training. G.A.K.

Effect of silicic acid on phagocytosis. E. ZANETTI AND L. PICCIOLI. *Med. del Lavoro*, **32**, 19–28, 39–45 (1941).—The admixture of fine silica dust in tiny doses to blood seems to first diminish and then to augment phagocytosis. Dissolved in blood, fine silica dust increases phagocytosis up to 130%. This increase is brought about by stimulation of the leucocytes which increases either the number of phagocytosed bacilli or the number of phagocytosing leucocytes. F.S.M.

Factors involved in the production and development of silicotic lesions. W. S. LEMON. *Radiology*, **37**, 413–20 (Oct., 1941).—Experimentally produced silicosis, like silicosis found among human beings, results from the interaction of particulate silica and the tissue-cell system of the lungs. The nature and degree of the defense reactions depend on the size, number, and mass of the particles of silica. The exact cause of the reaction is unknown, but it is probably brought about by electrochemical effects induced by contact of the particle and the cytoplasm of phagocytic cells. It probably proceeds because the laden cells are not killed but are preserved and may be concentrated into aggregations of cells situated along the course of the lymphatic system of the lungs. Arrest occurs as a result of phagocytosis and failure on the part of the eliminating mechanism of the lung. The pathognomonic criterion of experimentally produced silicosis is the discrete pseudotubercle, pearly white in color, glandular, subpleural or perivascular and peribronchial in situation, and visible and palpable on cut section. The morbid changes in lungs of experimental animals are controlled by encapsulation, calcification, and obliteration by collagenous and reticular fibers. Superimposed infection increases fibrosis in the perinodular parenchyma, causes ulceration and necrosis of bronchi and blood vessels, and brings about death of the animal by bronchitis, bronchopneumonia, and emphysema. Simple uninfected silicosis affects the function of the lungs in proportion to the amount of fibrous tissue that develops, with consequent reduction of vital capacity and a secondary loss of cardiac compensation. F.S.M.

Failure to provide modern dust-control equipment results in silicosis awards. ANON. *Occupational Hazards*, **4**, 32 (Oct., 1941).—Six cases involving disabilities resulting from silicosis, including Howard vs. Texas Owyhee Mining & Development Co. (Idaho) 115 Pac. 2nd 749 (July, 1941), were heard together, the facts in all of the cases being fundamentally the same. The claimants were miners employed in the company's mine. Operations included blasting as well as drilling. The company had installed wet-drilling apparatus, but much dust was present, nevertheless, and the claimants developed silicosis. Other than the wet-drilling equipment, the mining company had done little to improve conditions. Its failure to do so resulted in awards to the claimants. The Court stated that an injury caused by conditions which science and industry have learned to control and eliminate cannot be classed as an occupational disease. It stated that there is no cure for silicosis, but that hope in stemming the disease lies in preventive measures. The company had failed to provide any mechanical ventilation, had not installed exhaust pipes or filters, and had not supplied masks for the underground workers. The neglect to take these preventive measures resulted in a finding by the Court that the silicosis was not a noncompensable occupational disease under the Compensation Act but was a compensable injury. F.S.M.

Fatigue, its effects on industrial injuries. ANON. *Penna. Lab. & Ind. Rev.*, pp. 93–99 (July, 1941).—Industrial accidents occurring in 1940 in Pennsylvania are summarized. A rise in accidents with the number of hours worked, by different industries, is shown in tables. One peak is reached after 3 hr. of work, and another after 7 hr., indicating a close relationship to fatigue. By discovering the best time for rest periods for their particular kind of work, industries can profit by the attempt to prevent accidents. F.S.M.

Function of public health in defense. T. PARRAN. *Jour. Amer. Med. Assn.*, **117**, 186–88 (July, 1941).—The importance of public health efforts is evidenced by the figure of 350 million man-days lost last year owing to illness and accidents among workers, the equivalent of 1,000,000 men working a full year. This is a much greater loss than that caused by labor troubles. P. discusses the problem of men rejected for military service because of correctable defects and urges government aid in rehabilitating these men for use in industry. Nutrition problems, difficulties caused by the heavy influx of new workers to small communities, and the supply of physicians are also discussed. F.S.M.

High spots of the Midwest Power Conference. ANON. *Combustion*, **12** [11] 34–39 (1941).—The program included papers on power supply for national defense, power equipment defense trends, steam-turbine developments, supply of steam and interchange of power between a utility and industrial plants, forced-circulation boilers, federal hydro developments, variable-speed auxiliary drives, instruments and controls, and feed-water conditioning. H.E.S.

Hoods and piping in exhaust systems. C. A. SNYDER. *Occupational Hazards*, **3**, 8–9, 24 (Sept., 1941).—S. briefly discusses exhaust hood and piping design, listing twelve important points, some of which are as follows: use of forces created by the motion of the tool on the work to produce air currents into hoods; use of downdraft for heavy gases or dust and updraft for fumes, with cross or lateral draft being permitted for either; a ratio of 16 or less between the area of an exhaust hood and its exhaust pipe; double-walled hoods around shakeouts, etc.; simple adjusting devices; and ample access doors. S. states, "The most efficient dust collector is but half of a dust-control installation, and improper hood and piping designs can render the entire installation useless from an efficiency standpoint." See "Industrial . . . ," *Ceram. Abs.*, **19** [1] 34 (1940). F.S.M.

Industrial hygiene in the national defense program. J. J. BLOOMFIELD. *Ann. Internal Med.*, **15**, 165–71 (Aug., 1941).—Industrial mobilization in recent months has greatly augmented industrial-health problems. New workers are being taken on in increasing numbers, many of them not as physically fit as those they are replacing; unless the working environment is made safe for them, marked rises in accident and disease rate may be expected. The problem of fatigue will appear as a result of the speed-up in production. New and hazardous chemicals will come into use. Recent research and organization find us better

prepared to cope with these arising problems than ever before. A program has been outlined for the purpose of developing a nation-wide effort in industrial hygiene in defense industries. The responsibility of the medical profession in this program is discussed. F.S.M.

Industrial medical department: quarters and equipment. COUNCIL ON INDUSTRIAL HEALTH. *Jour. Amer. Med. Assn.*, **117**, 34–35 (July, 1941).—Floor plans for four rather elaborate industrial medical departments are presented. For as little as $150, a smaller medical department can be designed and equipped, but a more satisfactory department is obtained for $350. The general requirements are outlined, and a check list of equipment is shown. F.S.M.

Industrial medical service of the Standard Oil Co. of New Jersey and affiliated companies. ANON. *Jour. Amer. Med. Assn.*, **117**, 542–43 (Aug., 1941).—The Standard Oil Co. of New Jersey has maintained a medical service for its employees all over the world for 23 years. One hundred nine full-time physicians are employed, and, in the tropics, 32 hospitals with 1041 beds are operated. The service renders care to 186,344 employees. Placement and periodic physical examinations are emphasized; records are kept showing the causes for all disabilities lasting one or more days. Standard Oil Co. employees in this country average 6.31 days' disability a year, which is significantly lower than the average given for the country as a whole. Interesting differences are shown for U. S., European, and tropical employee groups. Sanitation and tropical diseases are being investigated, and a special study of 800 persons with hypertension is under way. F.S.M.

Industrial safety and health from an executive viewpoint. J. J. PORTER. *Ind. Med.*, **10** [12] 542–46 (1941).—P. refers to the medical research sponsored by the cement industry on the prevalence and dangers of silicosis in the industry. Cement and the raw materials from which it is made are high in lime, and any silica for the most part combines with lime, hence the popular misconception of silicosis as prevalent among cement workers. In 1934 the Committee on Accident Prevention of the North American Cement Corp. undertook a comprehensive study of the health of the workers. Eleven cement plants, geographically and otherwise representative of the industry, and 6 additional plants were selected for a qualitative and quantitative study of their various dusts. Of 35,000 workers ordinarily employed in cement manufacture in the U. S., 2278 men were studied for health and work environment, 1979 of whom were exposed to dust. The remaining 299, largely clerks, served as a control group to check the findings on those presumably exposed to dust. The research was conducted by the Saranac Laboratory under the direction of Leroy U. Gardner. The procedure and the check-over of the final reports are given. Results showed that silicosis is not ordinarily an occupational hazard in the industry. Only 8 men of the large number studied showed evidence of silicosis, and 6 of these had been exposed to silica dust in previous employment; the other 2 had been operating mills grinding sandstone. Conditions in these plants have been corrected, and no workmen are now exposed to silica dust in the cement industry. The survey also showed an unusually low incidence of other chronic respiratory diseases. K.R.

Medical plans for industry. K. ROBERTS AND M. W. BROWN. *Advanced Management*, p. 61 (April-June, 1941).—Absenteeism due to illness is costing industry more than $60 per employer per year. It is costing employees more. Strikes in 1940 resulted in a loss of about 2 hr. per worker per year; absenteeism due to illness resulted in a loss of approximately eight days per worker. The total of illness has been estimated at 400,000,000 man-days per year. Various means of reducing this high toll are discussed. Several plans are described and discussed in detail. The objectives of a proper program are threefold: (1) improved health of the working force; (2) security for the employee against the financial hazards of illness; and (3) increased efficiency and raised production level. F.S.M.

Medical technologist in industry. A. B. WEST. *Amer. Jour. Med. Tech.*, **7**, 76–81 (1941).—W. discusses the work of the medical technologist in large industrial plants, primarily with regard to blood counts, X rays of industrial diseases and injuries, and venereal disease programs. F.S.M.

Medical testimony in personal injury action. W. J. KERNAN. *Bull. N.Y. Acad. Med.*, **17**, 864–70 (Nov., 1941). F.S.M.

Noise in industry. C. PHARRIS. *Connecticut Health Bull.*, **55**, 254–55 (Sept., 1941).—Noise is not the least important among the various environmental conditions which do or may adversely affect the health of industrial workers. Too often noise is looked upon as an indispensable nuisance which must be tolerated. It has been found, however, that certain noisy occupations are conducive to hearing impairment. The influence of noise on the proficiency of workers has been graphically demonstrated, e.g., as the result of a reduction of 14.5% in the noise level in a large insurance office, the efficiency level was raised 8.8% and errors of typists and machine operators were reduced 29% and 50%, respectively. As a result, employee turnover dropped 47% and absenteeism dropped 37.5%. In another large office, the output of workers was increased 12% following a reduction of the noise level from 75 to 50 decibels. Preventive measures are briefly discussed. F.S.M.

Nomograph for exhaust-system design. B. KAGAN. *Heating & Ventilating*, **38**, 39–43 (Aug., 1941).—K. presents nomographs for calculating the resistance and air volume in an exhaust or blower system. The diagram facilitates especially the calculation of the air flow in a multiple-branch system where the resistance in the intersecting points must be balanced. F.S.M.

Ohio silicosis record. L. M. MERRITT. *Ohio Industrial Commission Monitor*, **14**, 126 (Aug., 1941).—Silicosis claims filed with the Industrial Commission up to July 31, 1941, are discussed. See "Silicosis . . . ," *Ceram. Abs.*, **20** [11] 276 (1941). F.S.M.

Principles of factory ventilation and heating. Application in wartime. T. BEDFORD. *Occupational Psychology*, **15**, 77–89 (April, 1941).—After considering the probable impurities in an ordinary workshop, B. recommends (1) adequate air movement, generally between 20 and 40 ft. per min., (2) variable air movement; (3) a room as cool as is compatible with comfort with a humidity not exceeding 70%; (4) an average temperature of the walls not appreciably lower than that of the air and preferably higher; and (5) warmer air at head level than at the floor. Some practical measures are suggested, e.g., warming tools the first hour or two of work when the room cannot be heated sufficiently. The effect of blackouts on ventilation is also considered. Natural ventilation is often seriously restricted by closing windows during blackouts, and remedies should be considered. F.S.M.

Profits in air: dust control. F. F. KRAVATH. *Heating & Ventilating*, **38**, 28–31 (June, 1941).—K. discusses the fundamentals of dust control and states, "We must fix it firmly in our minds that dust control is not something which we can buy quickly, easily, and painlessly from our favorite sheet-metal contractor. Dust control is not merely an exhaust system which can be purchased, run until it is completely worn out, outmoded, or inadequate, and then replaced with another exhaust system bought just as painlessly. Dust control is a living thing. It is a policy and a procedure devised and carefully checked by the plant's operating personnel and instituted and enforced by the plant's management whereby clean, healthful, dust- and fume-free atmospheric conditions are provided and maintained at all times within the plant." The same principle is applied to respirators and to medical examinations. Proper design of exhaust systems by the plant engineers, if possible, or by competent manufacturers is necessary before purchase and installation. The system must be designed to operate at all times when dust removal is necessary, not merely 75 or 90% of the time and operations. Visible dust, though often not as injurious as finer dust, must be eliminated, as workmen and courts are often im-

pressed by the extent of visible dust more than by the technicalities of dust counting. Strict regularity in frequency of dust counting should be observed, and dust counts at a distance from the plant give a comparison often clearer to courts and juries than the mere figures. The procedure following physical examinations of employees should be courageously conducted, i.e., men unfit for active work should be transferred to easier work and tuberculous employees should be isolated. Objections to this procedure on the part of either management or labor are shortsighted. **Electrostatic air filters.** H. W. POUND. *Ibid.*, pp. 34–37 (July, 1941).—Electrostatic filters remove dust particles that pass through mechanical filters, including those under 10 microns. An efficiency of 85% is reported for electrostatic filters at the normal air velocity of 500 ft. per min. compared with 20% for the best felt filter, using the U. S. Bureau of Standards discoloration test method. The efficiency rises to 95% at 300 ft. per min. Cases are cited in which improved products result from electrostatic air cleaning; in welding operations, recirculation of air without the expense of heating large quantities of outside air has been made possible. The Electro-Matic filter, combining electrostatic and mechanical filtration, is described briefly. **Wright Aeronautical Corporation's Lockland Plant—designer's views.** G. S. WHITAKER. **Owner's views.** H. E. LINSLEY. *Ibid.*, pp. 30–37 (Nov., 1941).—The air-conditioning system of the new plant, which is the largest single-story factory, is fully described. Of special interest is the automatic regulation of the entrance of outside air and its heating or cooling as the outside temperature varies. In the heat-treating section, the air is not cooled but washed and delivered at high humidity and high velocity into just the areas where the men are working. Individual air-conditioning units are used in small enclosed areas such as the first-aid room. F.S.M.

Revised occupational disease rating in Ohio. ANON. *Ohio Industrial Commission Monitor*, **14**, 118 (Aug., 1941).—The inclusion of silicosis and other diseases so increased the claim obligations that the flat two-cent occupational disease rate was producing inadequate premiums. To increase the disease premiums and also to provide an incentive for disease prevention in industries where disease hazard is prevalent, the law was amended to provide for a general disease rate of at least two cents for all classifications and, in addition, a higher disease rate in those classifications where the disease hazard is prominent. Occupational disease rates varying from a minimum of two cents in classifications with little or no indicated disease hazard to fifty cents where exceptionally high disease costs have developed have therefore been established. To indicate the disease portion included in the basic rate, a symbol specifying the amount of the disease portion is shown for those classifications where the disease rate is higher than two cents: oil distributors, coal mines (electrified), lime quarries, and bakeries, 10¢ extra; painting, coal mining (not otherwise classified), and planing mills, 15¢ extra; and brick manufacturing, structural-steel erection, and iron foundries, 20¢ extra. F.S.M.

Simple optical appliance for use with respirators. P. KINMONT. *Brit. Jour. Ophthalmol.*, **25**, 122–24 (March, 1941).—K. discusses the difficulties experienced with the usual procedures for adapting spectacles to the common breathing mask. A fitting devised for wear outside the mask eyepiece is described. The advantages of the device are discussed. F.S.M.

Substitution of ceramics for metals. HAROLD D. KUBE. *Bull. Amer. Ceram. Soc.*, **21** [1] 8–9 (1942).

Trends in occupational disease and in industrial hygiene. F. S. MALLETTE AND H. B. MELLER. *Ind. Med.*, **10** [11] 466–68 (1941).—During the 6 years from 1935 to 1940, industrial hygiene has shown its greatest, most rapid development, and much literature has been written on this subject. About 285 journals in this country and abroad covered pertinent material. By an actual numerical count of articles the four subjects receiving the most attention in the literature (1935–1940) are dust, lead, silicosis, and skin diseases. Climax years for interest in dust were 1937 and 1938, each year yielding 76 articles. In 1940 current interest in dust lessened to 46 articles. Silicosis has had relatively continuous interest focused upon it. Skin diseases show relative increase in published material. A marked drop in 1939 was followed by a sharp rise in 1940. Ventilation holds a definite interest level, with an increase in the literature in 1940. During the entire period, the number of articles devoted to the general subject of industrial hygiene practically doubled. 13 figures. K.R.

Trends in steam-plant development. A. G. CHRISTIE. *Combustion*, **12** [10] 35–38 (1941).—C. reviews the progress in the design of steam power-plant equipment to meet advances in steam conditions and operation, the influence on economy of generation, and some indications of future trends judged by experience with present practice. H.E.S.

Waste-heat boilers. J. B. CRANE. *Combustion*, **12** [7] 27–31 (1941).—C. discusses the types of boilers adapted to the recovery of heat from exhaust or from waste gases incident to industrial processes. Certain fundamental differences between waste-heat and fuel-fired boilers are pointed out, and typical calculations are included to illustrate the economics of waste-heat recovery. H.E.S.

What the nurse should know about industrial health hazards. E. A. WANNER. *Ohio Nurses Rev.*, **16**, 12–13 (Jan., 1941).—Besides actual duties in the plant, the nurse can be of great assistance to employer and employees through contact with the families of employees. Home conditions influencing the worker's efficiency which can be prevented can thus be discovered. The company's welfare work can be called to the attention of wives. The nurse must also lose no opportunity to emphasize the importance of receiving prompt treatment for injuries. F.S.M.

BOOKS AND SEPARATE PUBLICATIONS

Analyses of Skew Slabs. V. P. JENSEN. *Univ. Illinois Eng. Expt. Sta. Bull.*, No. 332, 110 pp. (1941). Price $1.00.—The behavior of skew slabs of reinforced concrete as used in modern highway and bridge construction is theoretically and experimentally investigated. Slab bridges having ordinary curbs, two or more lanes of traffic, spans of 20 ft. or less, and 45° skews are considered; theories are developed for deriving the actual load moments at different angles of skew and under different loads. Tables show the results of experiments on deflection and maximum loads for square, rectangular, and rhomboid-shaped slabs. M.H.

Fatigue of Workers and Its Relation to Industrial Production. NATIONAL RESEARCH COUNCIL COMMITTEE ON WORK IN INDUSTRY. Reinhold Publishing Corp., New York, 1941. 165 pp. Price $2.50.—This book will be useful to business executives, personnel workers, and all who have an interest in the increase of production at this important time. The purpose of this investigation was to determine the physical and mental reactions of industrial workers to the conditions to which they are exposed in their daily work. It reveals not only the direct causes of fatigue but also the psychological origin of strikes, slow downs, and voluntary limitation of output by workers. In addition to the working conditions in factories, several other types are considered, e.g., the effects of working at high altitudes, the influence of heat, etc. F.S.M.

Index to A.S.T.M. Standards Including Tentative Standards. American Society for Testing Materials, Philadelphia, Pa., 1941. 188 pp. Free. R.A.H.

Occupational Diseases. RUTHERFORD T. JOHNSTONE. W. B. Saunders Co., Philadelphia and London, 1941. 558 pp., 132 illustrations. Price $7.50.—While this book is intended primarily for the industrial physician, all who are interested in the field of industrial hygiene will find it interesting and valuable. The first three chapters present a succinct summary of the legal aspects of industrial medicine, a very useful condensation of scattered and voluminous material. Succeeding chapters deal with gases, solvents, fumes, metals, dusts, and miscellaneous topics. An extensive index is included. Illustrated. F.S.M.

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